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ORIGINAL PAPER

Spectrophotometric determination of Zn(II) ions using 6-bromo-4-hydroxy-3-nitrosonaphthalene-2,7-disulfonic acid

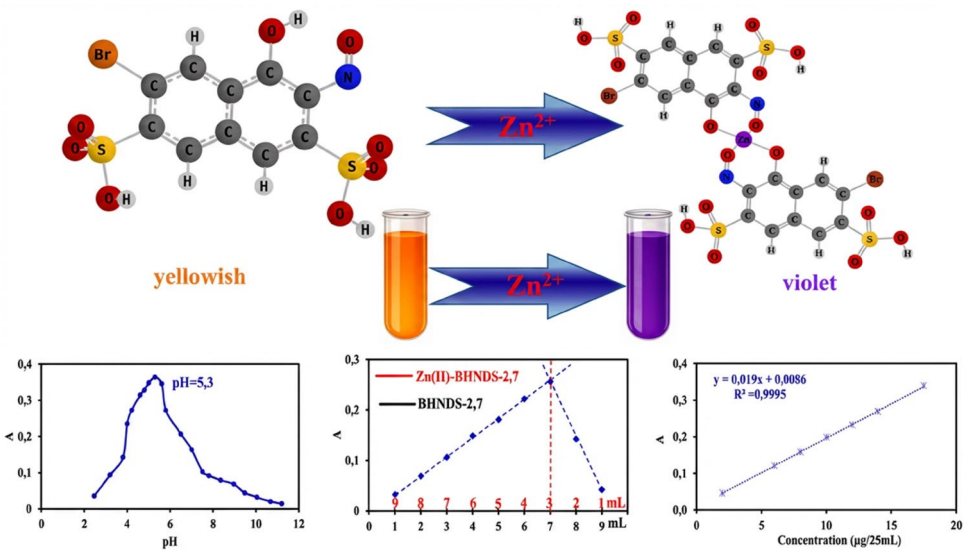
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Abstract

In this article, a new sensitive spectrophotometric method for the determination of Zn(II) using 6-bromo-4-hydroxy-3-nitrosonaphthalene-2,7-disulfonic acid is proposed. Optimal conditions were determined wavelength λ = 580 nm, solution pH 5.3, the amount of added reagent 1.2 mL, and stability over time was 8 h. Based on the selected optimal conditions, it was determined that the range of Beer's law is 1.0–17.5 μg/mL, Sandell's sensitivity is 0.001 μg/cm^2, and the contrast value due to the batochromic shift is Δλ = 85 nm. The reliability and reproducibility of the developed method were determined using the ''added-found'' method, according to which the relative standard deviation (Sr) did not exceed 1.66%, and the lower limit of determination (Q_{min}) was calculated to be 0.247 μg/25 mL. The developed method was successfully used to determine the content of zinc in the synthetic mixture and the ore of the Khandiza deposit, located in the Surkhandarya region of the Republic of Uzbekistan. The proposed method shows comparable or superior sensitivity, selectivity, and precision versus existing reagents and techniques, confirming its competitiveness for Zn(II) determination.

Graphical abstract



Keywords Zn(II) · 6-Bromo-4-hydroxy-3-nitrosonaphthalene-2,7-disulfonic acid (BHNDS-2,7) · Complex formation · Molar absorptivity

Extended author information available on the last page of the article

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Introduction

Zinc (Zn) plays an importante role in biological systems, is widely used in industrial processes, and is one of the most common trace elements in the environment (Kiouri et al. 2025). At the same time, excess amounts of Zn(II) ions can disrupt the ecological balance and negatively affect human health (Stiles et al. 2024). Therefore, the development of reliable, sensitive, and selective analytical methods for determining Zn(II) ions in natural, industrial, and biological objects is of great scientific and practical importance (Liu et al. 2025; Zheng et al. 2020).

Different analytical methods, such as atomic absorption spectroscopy, voltammetry, and spectrophotometry, are used to determine Zn(II) ion (Al-Murshedi et al. 2025; Aydinoglu 2022; Bazrafkan et al. 2025; Kawthar Abed Al Ameer and Ibtehaj Raheem Ali 2025; Radhi 2024; Vlasova et al. 2025). These include spectrophotometric methods, which are distinguished by simplicity, cost-effectiveness, and sufficient sensitivity(Passos and Saraiva 2019).

In particular, methods developed based on chromogenic reagents that form colored complexes with intermediate metal ions are widely used for detection at very low concentrations (Tavancheh and Beiraghi 2020).

A spectrophotometric method for the determination of Zn(II) ions using N-(2-(2-amino-5-nitrophenyl)diazenyl) ethyl)naphthalen-1-amine as a chromogenic reagent has been reported. The Zn(II) complex exhibited maximum absorbance at 468 nm with a metal-to-ligand ratio of 1:1 and remained stable for 60 min. Beer's law was obeyed in the concentration range of 0.653–6.538 μg mL^{-1} ($R^2 = 0.9997$). The molar absorptivity was $8.74 \cdot 10^3$ L mol^{-1} cm^{-1}, while the limits of detection and quantification were 0.096 and 0.291 μg mL^{-1}, respectively. The method showed a Sandell sensitivity of 0.011 μg cm^{-2}, recoveries of 98.48–100.00%, relative standard deviations of 0.031–0.127%, and errors ranging from − 1.75 to 0.25%, indicating good sensitivity, precision, and accuracy (Abduljaleel et al. 2025).

A spectrophotometric method based on murexide as a chromogenic reagent has been developed for the determination of Zn(II) and Cu(II) ions. The Zn(II)–murexide and Cu(II)–murexide complexes exhibited maximum absorbance at 450 and 470 nm, respectively, with a metal-to-ligand ratio of 1:2. The proposed method enabled the determination of Zn(II) and Cu(II) in the concentration ranges of 0.2–2.0 and 0.5–5.0 ppm, respectively, with molar absorptivities of $1.95 \cdot 10^4$ and $6.55 \cdot 10^3$ L mol^{-1} cm^{-1}. The formed complexes showed high stability over a wide pH range, allowing the simultaneous determination of both metal ions (Muftah Elsherif et al. 2022). A spectrophotometric method for Cr(III) determination using sodium 4-((4,5-diphenyl-imidazol-2-yl)diazenyl)-3-hydroxynaphthalene-1-sulfonate (SDPIHN) in the presence of Triton X-100 has been reported. The Cr(III) complex exhibited maximum absorbance at 586 nm and remained stable for more than 24 h at pH 10. Beer's law was obeyed in the concentration range of 1–11 μg mL^{-1}, with a molar absorptivity of $2.44 \cdot 10^3$ L mol^{-1} cm^{-1}, Sandell sensitivity of 0.021 μg cm^{-2}, and LOD and LOQ values of 0.117 and 0.385 μg mL^{-1}, respectively. Stoichiometric studies revealed a metal-to-ligand ratio of 1:3, while the method demonstrated satisfactory precision and accuracy based on RSD and relative error values (Ameer and Hussain 2023).

However, studies presented in the literature(Gavazov et al. 2024; Madatov et al. 2024; Pavun et al. 2024) show that many reagents are limited by insufficient selectivity, sensitivity to the action of foreign ions, and the inability to form stable complexes.

In recent years, analytical organic reagents containing functional groups, particularly hydroxyl and nitroso groups, have been widely studied as promising chromogenic reagents due to the formation of strong chelate-type complexes with metal ions based on a donor–acceptor mechanism (Li et al. 2025; Shi et al. 2021). Nevertheless, the issue of developing analytical methods with high selectivity and sensitivity for determining Zn(II) ions based on such compounds remains relevant, and systematic and in-depth research in this direction has not been conducted to a sufficient extent (Gholipour et al. 2025; Khan et al. 2024; Rus et al. 2022).

From this point of view, the BHNDS-2,7 reagent is a promising chromogenic analytical organic reagent for the determination of Zn(II) ions. However, the mechanism, stability, and analytical properties of its complexation with Zn(II) have not been sufficiently studied, and systematic data on this matter are lacking.

Therefore, the novelty of the present work lies in the first systematic investigation of the Zn(II)–BHNDS-2,7 complex and the development of a new spectrophotometric method for Zn(II) determination based on this reagent. The study provides comprehensive information on the optimal conditions of complex formation, stoichiometry, stability, and analytical performance of the Zn(II)–BHNDS-2,7 system, as well as elucidates the complexation mechanism using UV–Vis, IR, and NMR spectroscopic techniques.

In this work, a sensitive and selective spectrophotometric method for determining Zn(II) ions using the reagent BHNDS-2,7 was developed. The proposed method possesses high sensitivity and selectivity compared to existing methods and can be effectively used to detect Zn(II) ions in real objects of complex composition, including environmental and industrial samples.

Experimental

Reagents

Throughout the research, analytical reagents were used. Zinc(II) solutions were prepared from $Zn(NO_3)_2 \cdot 6H_2O$ (CAS No. 10196-18-6). The chromogenic reagent 6-bromo-4-hydroxy-3-nitrosonaphthalene-2,7-disulfonic acid (CAS No. 212814) was used for complex formation. All chemicals were obtained from Sigma-Aldrich and used without further purification. Boric acid, acetic acid, phosphoric acid, and bidistilled water were used as available in the laboratory without further purification.

Characterization

Spectrophotometric measurements of the colored solutions were carried out using an EMC-31PC-UV spectrophotometer (Germany). The pH of the solutions was measured using a pH-150 M pH meter (OAO "Gomel Plant of Measuring Instruments", Belarus) with an accuracy of ± 0.05 pH units. IR spectral analysis was performed using a Bruker spectrometer (Germany). ^{1}H NMR spectra were recorded on an Avance III HD 400 NMR spectrometer (Germany).

Preparation of solutions and calibration procedures

4.3981 g of precisely weighed crystalline salt was dissolved in bidistillate water in a 1000 mL volumetric flask to prepare a stock solution of 1 mg/mL. Working solutions were obtained by sequentially diluting the stock solution to the required concentration range before each experiment. The reagent solution was prepared by dissolving 0.0500 g of BHNDS-2,7 in distilled water and diluting to the mark in a 100 mL volumetric flask. Universal buffer solutions were prepared by mixing the corresponding ratios of boric acid (0.04 M), phosphoric acid (0.04 M), and acetic acid (0.04 M) in the pH range of 2.46–11.20, and then equalized with a sodium hydroxide solution (0.2 M).

Determination method: a volumetric flask of 25 ml was filled with 1.2 ml of 0.05% BHNDS-2,7 reagent solutions, 5 ml of universal buffer solutions with a pH value of pH = 5.3, and 5 µg/ml of alternating amounts of Zn (II) ion-containing solutions, diluted with bidistillate water up to the flask mark. The absorbance of the resulting complex solution was measured at 580 nm using an EMC-31PC-UV spectrophotometer against a reagent blank over a fixed time interval.

Results and discussion

Investigation of the appropriate experimental conditions

Zinc(II) ions exhibit a pronounced tendency to form stable coordination complexes with organic ligands containing oxygen- and nitrogen-donor atoms (Kargar et al. 2021). The reagent BHNDS-2,7 was selected due to its high chelating capacity through hydroxyl (–OH) and nitroso (–NO) functional groups, leading to the formation of an intensely colored and stable Zn(II)–BHNDS-2,7 complex with a stoichiometric ratio of 1:2 (metal:ligand), suitable for sensitive spectrophotometric determination Fig. 1.

In spectrophotometric analysis methods, the sensitivity and selectivity of determination largely depend on the optimal conditions of reaction (Mishra et al. 2026). Specifically, the process of complexation of metal ions with chromogenic reagents significantly depends on the solution medium, pH, buffer solution composition, stability over time, the pouring order of the initial components, reagent concentration, and other factors (Liao et al. 2022). Therefore, the selection of

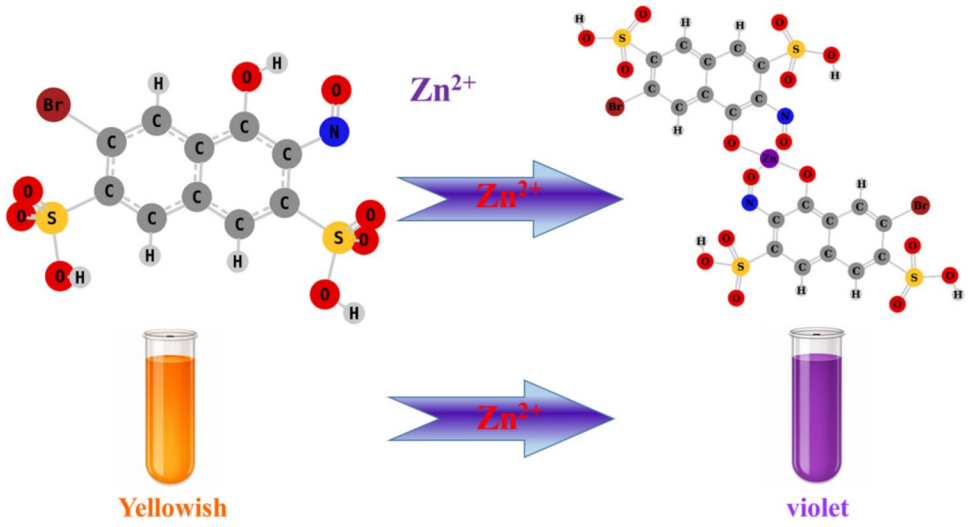


Fig. 1 Scheme of the Formation of the Zn(II)–BHNDS-2,7 Complex

optimal conditions is an important stage in the development of the analytical method.

Taking into account that one of the important conditions for the implementation of complexation reactions is the solution medium, universal buffer solutions with a pH of 2.46–11.20 were prepared to select optimal conditions for complex compounds of Zn (II) ions with the reagent BHNDS-2.7. According to the experimental results Fig. 2a, the maximum optical density of the complex compound was observed in the pH region of 5.0–5.6. An optimal pH of 5.3 was selected. This behavior can be explained by the protonation–deprotonation equilibrium of the donor centers of BHNDS-2.7. At pH 5.3, partial deprotonation of the hydroxyl group enhances its coordination ability toward Zn(II), while the nitroso donor center remains available for complex formation. At the same time, hydrolysis of Zn(II) is minimized, providing favorable conditions for stable chelate formation. To investigate the dependence of the buffer solution composition on the main reaction components, universal, sodium citrate, acetate, and sodium hydrophthalate buffer solutions were prepared for the Zn (II) ion at pH = 5.3. According to the results presented in Fig. 2b, under the influence of a universal buffer solution, the colored complex exhibits the highest optical density. The universal buffer maintains a stable proton concentration and therefore ensures the optimal protonation state of the ligand donor atoms, resulting in maximum complex formation and absorbance.

During the research, the stability of complex formation over time was studied. The fact that the optical density of the zinc complex remained unchanged for up to 8 h indicates that the complex is highly stable, and this time is also considered sufficient for analysis. Furthermore, the order of addition of the initial components was investigated, and the reagent–buffer–Zn(II)–distilled water sequence was selected as the optimal procedure. This sequence likely provides favorable conditions for complex formation by ensuring the proper protonation state of the ligand before contact with Zn(II), thereby facilitating rapid equilibration and minimizing competing side reactions such as metal hydrolysis or non-specific interactions in solution.

The reagent concentration is selected in sufficient excess to fully ensure the equilibrium in complex formation, but its excess amount can increase background absorption (Ait Khaldoun et al. 2026). Therefore, the effect of reagent concentration was studied Fig. 2c. According to the results of the study, it was established that the amount of 0.05% BHNDS-2,7 at a concentration of 1.2 mL is sufficient compared to $T_{Zn}^{2+} = 10$ µg/25 mL.

The UV–Vis absorption spectra of BHNDS-2,7 and its Zn(II) complex are presented in Fig. 2d. The free reagent exhibited a maximum absorption band at 495 nm, whereas the Zn(II)–BHNDS-2,7 complex showed an intense band at 580 nm. The appearance of this new band, together with the bathochromic shift of the absorption maximum toward a longer wavelength (Δλ = 85 nm), indicates the formation of

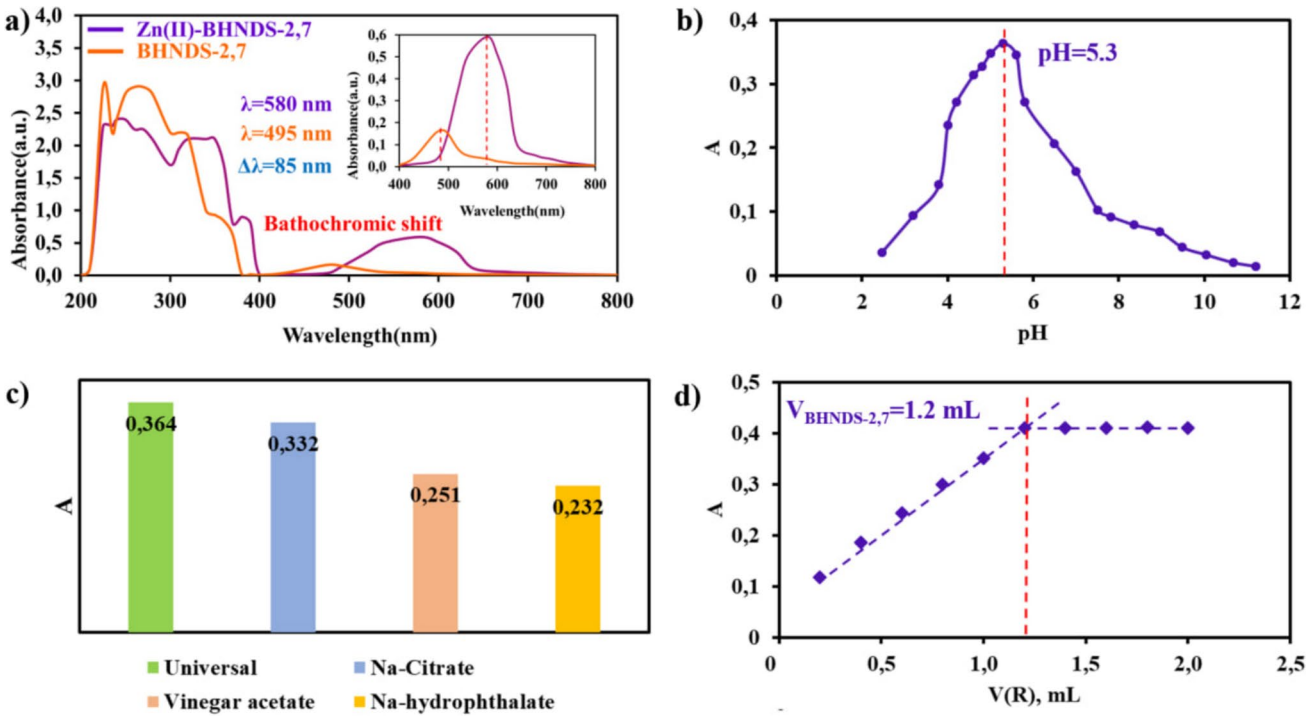


Fig. 2 Effect of pH (**a**), buffer composition (**b**), and reagent amount (**c**) on the absorbance of the Zn(II)–BHNDS-2,7 complex; absorption spectra of BHNDS-2,7 and its Zn(II) complex (**d**)

a stable complex and reflects changes in the electronic structure of the ligand upon coordination with Zn(II). Intense absorption bands observed in the 230–300 nm region for both the reagent and the complex are attributed to $\pi \rightarrow \pi^*$ transitions of the aromatic chromophore system (Bhosale et al. 2025; Cao et al. 2025; Giang et al. 2025; Nagaraj et al. 2025). The absorption band near 495 nm for the free ligand is assigned to $n \rightarrow \pi^*$ transitions involving donor atoms of the chromogenic reagent. Upon coordination with Zn(II), this band shifts to 580 nm owing to metal–ligand interaction and charge-transfer effects. The observed bathochromic shift can be attributed to electron density redistribution and enhanced π-electron delocalization within the chelate system formed through coordination of Zn(II) with the donor centers of BHNDS-2,7. Since Zn(II) possesses a filled d^{10} electronic configuration, classical d–d transitions are not expected; therefore, the spectral changes are mainly associated with ligand-centered transitions and coordination-induced charge-transfer interactions. The pronounced bathochromic shift and increased absorbance at 580 nm confirm complex formation and provide the analytical basis for the spectrophotometric determination of Zn(II).

The Sandell sensitivity of the method, defined as the amount of substance (μg/mL) corresponding to an absorbance of 0.001, was calculated using the following formula (Al-Taee and Al-Enizzi 2024):

$$\text{S.s.} = \frac{Q \cdot l \cdot 0.001}{\text{A} \cdot 25} = \frac{10 \cdot 1 \cdot 0.001}{0.5910 \cdot 25} = 0,001 \ \ \mu\text{g/mL} \qquad (1)$$

Here, S.s. is the Sandell sensitivity, Q is the amount of the metal ion, and A is the optical density (absorbance).

As seen from the results in Table 1, the reaction has a slight contrast (Δλ = 85 nm) and high sensitivity (S.s. = 0.001 μg/cm^2), which indicates that the method is effective and selective.

BHNDS-2.7-to-Zn(II) molar ratio

In the course of the research, the active form of the Zn(II) ion in complex formation was identified based on the Nazarenko method. The results indicated that the free Zn^{2+} ion is the reactive species participating in the complexation process. Under the selected experimental conditions (pH 5.3), hydrolysis of Zn(II) is negligible, and Zn^{2+} remains the predominant form in solution. The composition and stoichiometry of the resulting complex were systematically studied using different physicochemical approaches. Specifically, the dependence of optical density on the component ratio was analyzed using the Job method Fig. 3a, the linear dependence of the analytical signal was determined using the linear Asmus method Fig. 3b, and the results obtained by spectrophotometric titration were verified Fig. 3c. The obtained results are consistent with each other and reliably confirm that the molar ratio of the components in the complex is Zn(II):BHNDS-2.7 = 1:2.

Analytical characteristics

In order to comprehensively study the process of complex formation of the Zn(II) ion with the reagent BHNDS-2.7, the main analytical and thermodynamic characteristics of this complex were determined. In particular, the actual molar absorptivity coefficient of the Zn(BHNDS-2,7)$_2$ complex (ε_{real} = 51,282 L mol^{-1} cm^{-1}) and the equilibrium constant of complex formation (K_{eq} = 0.03) were calculated using Tolmachev's graphical method Fig. 3d. The high molar absorptivity value indicates an intense electronic transition and is consistent with ligand-centered transitions accompanied by coordination-induced charge-transfer interactions. Considering the filled d^{10} electronic configuration of Zn(II), classical d–d transitions are not expected in the complex. The substantially higher molar absorptivity of the Zn(BHNDS-2,7)$_2$ complex compared to the free reagent enhances photometric sensitivity and spectral contrast, enabling reliable trace-level determination of Zn(II). The obtained Keq value reflects the influence of protonation–deprotonation equilibria on the complexation process under the selected experimental conditions.

$$\varepsilon_{haq} = \frac{1}{1/\varepsilon \cdot 10^n} = \frac{1}{1,95 \cdot 10^{-5}} = 51282 \qquad (2)$$

$$K_{muv} = \frac{C_H^n \cdot l^n}{n^n \cdot \varepsilon_x \cdot \beta^{n+1}} = \frac{(5,0 \cdot 10^{-6})^2 \cdot 1^2}{2^2 \cdot 51282 \cdot (1,532 \cdot 10^{-5})^3} = 0,03 \qquad (3)$$

Here, C_H–H^+ is the concentration of hydrogen ions, ℓ is the optical path length (cuvette thickness), ε_{real} is the true molar absorptivity of the complex, and n is the stoichiometric coefficient.

The stability constant of the complex (logβ = 18.96) was determined using the Babko's dilution method Table 2. The

Table 1 Spectral characteristics of the BHNDS-2,7 and the Zn(II)-BHNDS-2,7 complex

Complex color	pH	λ_{max}, nm		Δλ	$C_{Zn^{2+}}$, μg /25 mL	$C_{Zn^{2+}}$, (mol/L)	$\bar{A}$	Sandell sensitivity, μg/cm^2
		Zn(II)	BHNDS-2.7					
Zn(II)-BHNDS-2,7 (n = 5)								
Violet	5.3	580	495	85	15	$9.176 \cdot 10^{-6}$	0.5910	0.0010

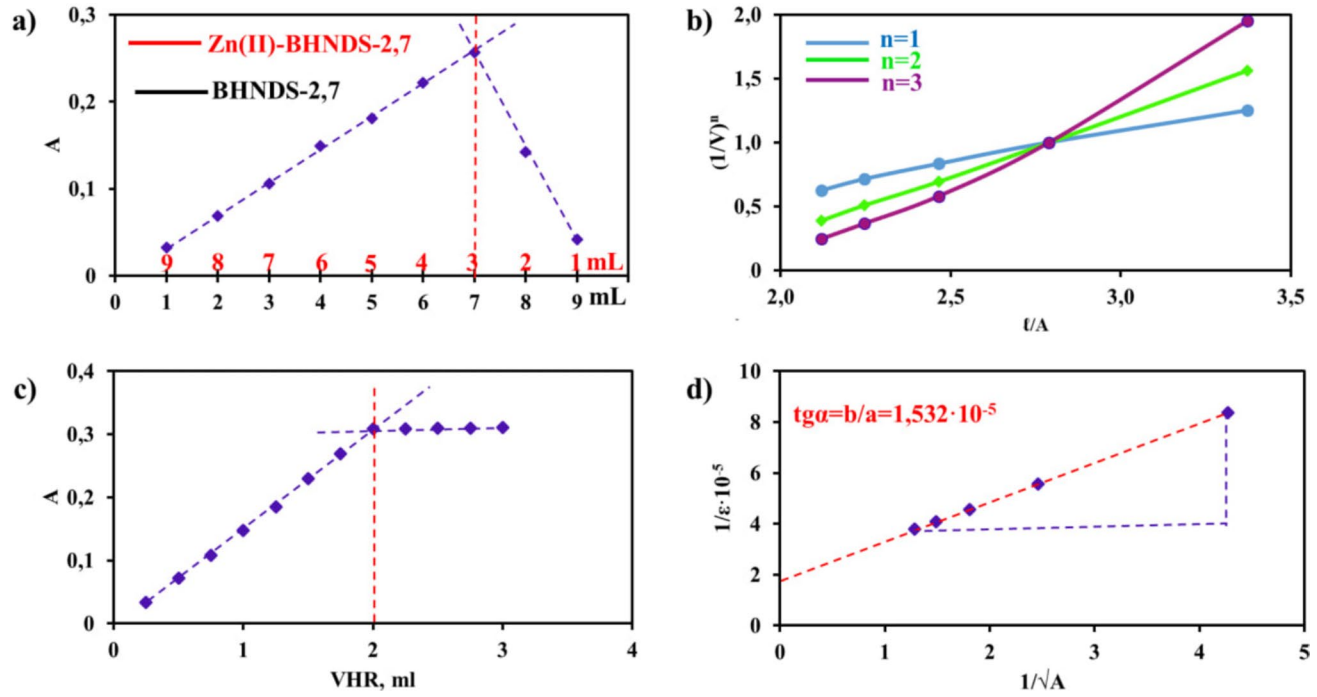


Fig. 3 Graphs of Job's method (**a**), the Asmus straight-line method (**b**), the spectrophotometric titration method (**c**), and determination of molar absorptivity (**d**)

Table 2 Results of determining the stability constant of the complex using the dilution method

№	$V_{Zn^{2+}}$. mL	V_{R1}. mL	$C_1 \cdot 10^{-6}$	A_1	$C_2 \cdot 10^{-6}$	A_2	$K_{unstable\ (MeR)}$	$K_{unstable\ (MeR)}$ (β_k)	$lgK_{stable\ (MeR)}$($lg\beta_k$)
1	0.5	1	4.59	0.0550	2.295	0.0270	$2.14 \cdot 10^{-21}$	$4.67 \cdot 10^{20}$	20.67
2	1.0	2	9.18	0.1650	4.590	0.0800	$5.67 \cdot 10^{-20}$	$1.76 \cdot 10^{19}$	19.25
3	1.5	3	13.8	0.3010	6.900	0.1430	$5.64 \cdot 10^{-19}$	$1.77 \cdot 10^{18}$	18.25
4	2.0	4	18.4	0.4510	9.200	0.2180	$3.01 \cdot 10^{-19}$	$3.32 \cdot 10^{18}$	18.52
5	2.5	5	22.9	0.6060	11.45	0.2910	$7.84 \cdot 10^{-19}$	$1.28 \cdot 10^{18}$	18.11
Σ							$3.42 \cdot 10^{-19}$	$9.82 \cdot 10^{19}$	18.96

Table 3 Results of the determination of the dissociation constant and molar absorptivity of the BHNDS-2,7 reagent by Komar's method

No	pH	Color	A_i					
			A_1	A_2	A_3	A_4	A_5	$\bar{A}$
1	3	Reddish	0.0810	0.0820	0.0820	0.0830	0.0820	0.0820
2	7	Pink	0.1020	0.1030	0.1040	0.1030	0.1040	0.1030
3	10	Light purple	0.0710	0.0720	0.0710	0.0730	0.0710	0.0720

high stability constant indicates the formation of a thermodynamically stable Zn(II)–BHNDS-2,7 chelate, which contributes to the sensitivity and selectivity of the proposed spectrophotometric method. Furthermore, the dissociation constant ($K_{diss} = 6.71 \cdot 10^{-8}$) and the molar absorptivity coefficient ($\varepsilon = 2704$ L mol^{-1} cm^{-1}) of the BHNDS-2.7 reagent were determined using the Komar method in the Table 3. The obtained dissociation constant indicates that the protonation state of the BHNDS-2,7 reagent is strongly pH-dependent. At the optimal pH of 5.3, partial deprotonation of the coordinating groups provides favorable conditions for interaction with Zn(II), while excessive hydrolysis of the metal ion is avoided. This balance contributes to efficient complex formation under the selected experimental conditions (Demchuk et al. 2025).

$$K_{unstable.(MeR)} = \frac{[H^+] \cdot (A_1 \cdot C_2 - A_2 \cdot C_1)^{q+1}}{(C_1 \cdot \sqrt[3]{A_2} - C_2 \cdot \sqrt[3]{A_1}) \cdot (A_1 \cdot \sqrt[3]{A_2} - A_2 \cdot \sqrt[3]{A_1})^q} \quad (4)$$

$$K_{stable.(MeR)} = \frac{1}{K_{unstable.(MeR)}} \quad (5)$$

Here: $[H^+]$ is the concentration of hydrogen ions, A_1, A_2 A_3 are optical densities, and C_1, C_2, C_3 are metal ion concentrations.

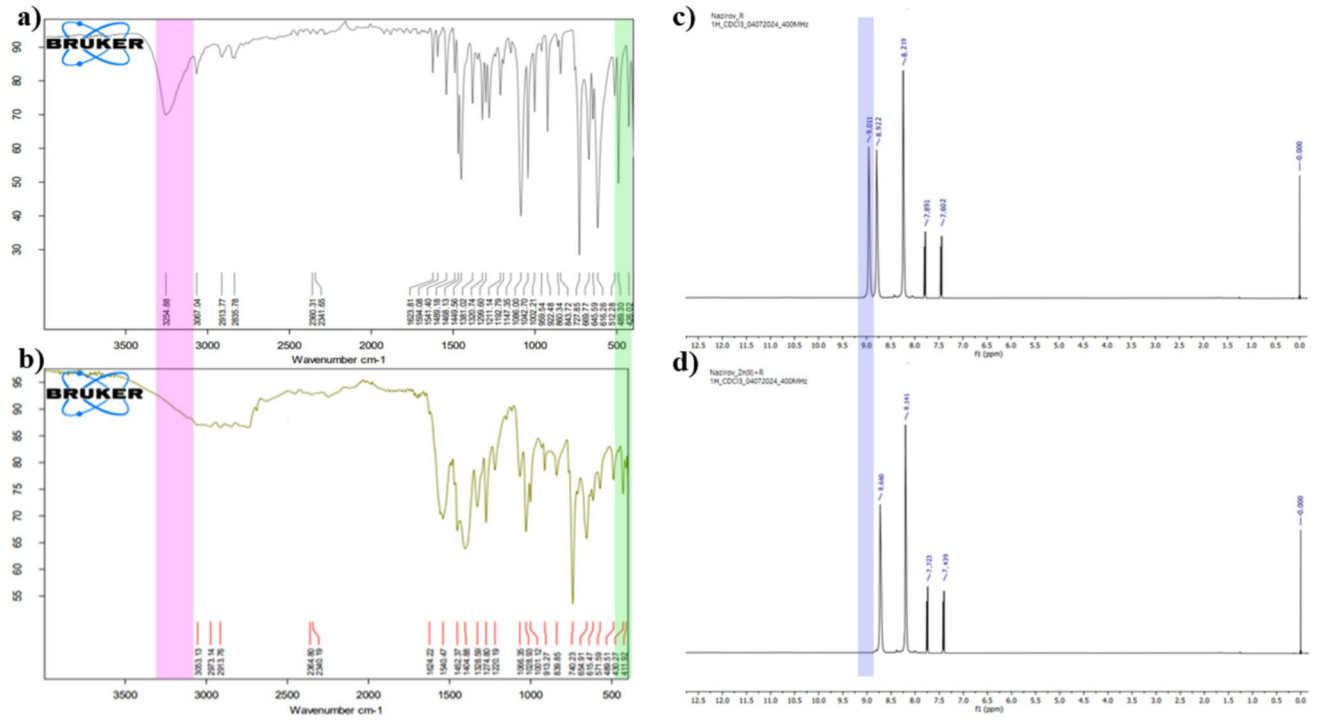


Fig. 4 IR and ^{1}H NMR spectra of BHNDS-2,7 (**a**, **c**) and Zn(II)–BHNDS-2,7 complex (**b**, **d**)

$$K_{diss(HR)} = \frac{(h_1 - h_2)(A_1h_1 - A_3h_3) - (h_1 - h_3)(A_1h_1 - A_2h_2)}{(A_1 - A_2)(h_1 - h_3) - (A_1 - A_3)(h_1 - h_2)} = 6,71 \cdot 10^{-8} \quad (6)$$

$$\varepsilon_{\mathrm{HR}} = \frac{1}{C \cdot \ell} \cdot \frac{(A_1 - A_2)(A_1h_1 - A_3h_3) - (A_1 - A_3)(A_1h_1 - A_2h_2)}{(A_1 - A_2)(h_1 - h_3) - (A_1 - A_3)(h_1 - h_2)} = 2704 \quad (7)$$

here: h_i is the concentration of hydrogen ions in the solution, mol L^{-1}; A is the optical density, C is the total concentration of the reagent, mol·L^{-1}; ℓ is the thickness of the absorbing layer (1.0 cm).

Spectral properties of ligand and complex

The structure of the reagent BHNDS-2,7 and the zinc complex was studied using IR spectroscopy. Spectral analysis was performed using the ‘‘BRUKER” technical instrument. The sample taken for analysis was solidified and converted into tablets using potassium bromide. The analysis results were obtained at 400–4000 cm^{-1} Fig. 4a, b.

Based on the results in Table 4, it was confirmed that in the IR spectrum of the BHNDS-2.7 reagent, the stretching vibrations νs and deformation vibrations δ of the hydroxyl group ν (OH) with a weak hydrogen bond disappeared at 3254.88 cm^{-1} and 1211.14 cm^{-1}, and a new peak belonging to the O-Zn bond was formed at 499.84 cm cm^{-1}. At the same time, it can be concluded from the results of the shift

Table 4 Characteristic absorption frequencies in the IR spectra of BHNDS-2,7 and the Zn(II)–BHNDS-2,7 complex

Vibrational modes	BHNDS-2.7	Zn(II)-BHNDS-2.7	
ν(OH)	3254.88	-	cm^{-1}
δ(OH)	1211.14	-	
ν(N=O)	1623.81	1676.10	
ν_s(C–N)	1086.00	1068.36	
ν_{as}(C–N)	860.34	882.82	
ν (C–H)	3067.04	3027.26	
ν_s (C–H)	2913.77	2922.74	
δ(C–H)	1381.02	1325.10	
ν_{as}(C–H)	2835.78	2841.70	
ν(S=O)	959.54	967.49	
ν(S–O)	843.72	825.65	
ν(SO_3H)	1299.6	1279.10	
ν(C–Br)	645.59	668.75	
Naphthalene ring	727.85	744.04	
O–M	-	499.84	

of peaks belonging to the valence vibration frequency of the (N=O) group that Zn(II) ions can form complex compounds with a Zn(II): BHNDS-2,7 ratio of 1:2 by forming a simple bond and a donor–acceptor bond with the nitroso(N=O) group due to the replacement of the hydrogen atom in the hydroxyl group ν(OH) of the BHNDS-2,7 reagent.

According to the BHNDS-2.7 spectrum obtained by the Avance III HD 400 NMR spectrometer, the H atoms in the 1C-bonded OH group located in the naphthalene ring, the H atoms in the 8C-bonded H, and the H atoms in the OH sulfo group are located in an intensive singlet state at

9.011 ppm, 8.922 ppm, and 8.219 ppm. H atoms bonded to 4C and 5C atoms signaled in a doublet state at 7.891 ppm and 7.602 ppm Fig. 4c. The results of the NMR spectrometry of the complex compound formed by the Zn(II) ion with the BHNDS-2,7 reagent in Fig. 4d show that the H atoms in the 8C-bonded H and the OH atoms in the sulfo group are located in an intensive singlet state at 8.660 ppm and 8.141 ppm. Atoms of H bonded to 4C and 5C atoms signaled in a doublet state at 7.723 ppm and 7.439 ppm. The loss of an intensive singlet signal belonging to an H atom in the OH group bound to 1C located in the naphthol ring indicates that it has been replaced by a Zn ion. The observed shifts of the proton signals to lower chemical shift values after complexation indicate a redistribution of electron density within the ligand upon coordination with Zn(II). These changes, together with the disappearance of the 1C–OH proton signal, further confirm the involvement of the hydroxyl group in the complex formation process.

The Asmus straight-line method, the Job method, the spectrophotometric titration method, and UV–Vis, IR, and NMR-1H spectroscopy methods studied during the study confirmed each other. In conclusion, it can be stated that Zn(II):BHNDS-2,7 corresponded to a molar ratio of 1:2. The coordination of two bidentate BHNDS-2,7 ligands through hydroxyl oxygen and nitroso donor centers suggests a tetrahedral geometry around the Zn(II) ion, which is consistent with the coordination preferences of d^{10} Zn(II) and the high stability constant (logβ = 18.96) of the complex. The structure of the $Zn(BHNDS\text{-}2,7)_2$ complex compound is proposed.

Applicability of Beer's law and sensitivity

A volumetric flask with a volume of 25 mL was diluted with 1.2 mL of a 0.05% BHNDS-2.7 reagent solution, 5 mL of a universal buffer solution with a pH of 5.3, and 5 µg/mL of a solution containing Zn(II) ions in variable amounts, followed by the addition of bidistillate water to the flask mark. The optical density of the complex compound solutions was measured on an EMC-31PC-UV spectrophotometer at a wavelength of λ = 580 nm for the Zn(II) ion relative to the reference solution at a specific time interval. The results obtained are presented in Fig. 5a.

According to the results obtained, the domain of the Beer law is observed in the range of 1.0–17.5 µg/mL. At higher concentrations, there was a deviation from the linear relationship. Such deviations may be associated with changes in solution equilibrium and non-ideal absorption behavior at elevated concentrations.

Regression parameters (a = 0.0086 and b = 0.019) and the coefficient of determination ($R^2 = 0.9995$) were calculated using the least squares method. The results obtained indicate a strong linear relationship between the analytical signal and the concentration, characterizing the calibration graph with high accuracy (Pagliano et al. 2015) (Table 5, Fig. 5b, and Eqs. 8–16).

$$a = \frac{\Sigma xi^2 \cdot \Sigma yi - \Sigma xi \cdot \Sigma xi \cdot yi}{n \cdot \Sigma xi^2 - (\Sigma xi)^2} = \frac{850,25 \cdot 1,366 - 69,5 \cdot 16,572}{7 \cdot 850,25 - 69,5^2} = 0,0086; \tag{8}$$

$$Sy = \sqrt{\frac{\Sigma(i - Y_{hisob.})^2 \cdot 10^n}{n-2}} = \sqrt{\frac{29,1 \cdot 10^{-6}}{7-2}} = \sqrt{\frac{29,1 \cdot 10^{-6}}{5}} = 0,0011; \tag{9}$$

$$Sa = Sy\sqrt{\frac{\Sigma xi^2}{n \cdot \Sigma xi^2 - (\Sigma xi)^2}} = 0,0011\sqrt{\frac{850,25}{7 \cdot 850,25 - 69,5^2}} = 0,0009; \tag{10}$$

$$tg\alpha = \frac{a}{Sa} = \frac{0,0086}{0,0009} = 9,2; \tag{11}$$

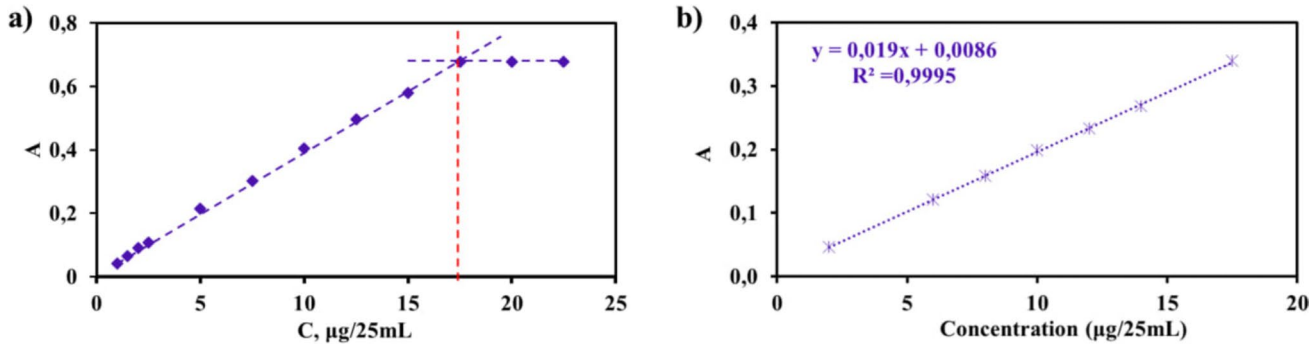


Fig. 5 Graphs of the Beer law dependence (**a**) and the calibration curve for Zn(II) determination (**b**)

Table 5 Calibration curve parameters and results of data processing

No.	Zn(II) µg. Xi	Ā. Yi	Xi^2	Xi ·Yi	$Yi_{account}$	$Y–Yi_{account}$	$(Yi–Yi_{account.})^2 . 10^{-6}$
1	2	0.0470	4	0.094	0.046	0.001	0.628
2	6	0.1210	36	0.726	0.121	0.000	0.120
3	8	0.1580	64	1.264	0.159	-0.001	0.838
4	10	0.1990	100	1.990	0.196	0.003	6.33
5	12	0.2330	144	2.796	0.234	-0.001	1.11
6	14	0.2680	196	3.752	0.272	-0.004	13.1
7	17.5	0.3400	306.25	5.950	0.337	0.003	6.92
∑	69.5	1.3660	850.25	16.572	1.366		29.1

Table 6 Results of the accuracy and precision of the Zn(II) ion determination method with the BHNDS-2.7 reagent (n=3, P=0.95)

No.	Zn(II) added. µg	$A_{1.2.3}$	Found Zn(II). µg	$\overline{X}_i$	$X_i - \overline{X}$	$(X_i - \overline{X})^2$	S	Sr%	ΔX	X±ΔX	Relative error (%)
1	2	0.0464	1.99	1.99	0.00	0.0000	0.030	1.51	0.075	1.99±0.07	0.50
		0.0468	2.02		0.03	0.0009					
		0.0458	1.96		-0.03	0.0009					
2	6	0.1230	6.02	5.99	0.03	0.0009	0.031	0.52	0.077	5.99±0.078	0.17
		0.1222	5.98		-0.01	0.0000					
		0.1219	5.96		-0.03	0.0009					
3	8	0.1620	8.07	7.98	0.09	0.0081	0.090	1.13	0.224	7.98±0.22	0.25
		0.1605	7.99		0.01	0.0001					
		0.1586	7.89		-0.09	0.0081					
4	10	0.2000	10.07	9.97	0.10	0.0100	0.091	0.91	0.226	9.97±0.23	0.30
		0.1980	9.97		0.00	0.0000					
		0.1960	9.86		-0.11	0.0121					
5	12	0.2380	12.07	11.95	0.12	0.0144	0.108	0.90	0.268	11.95±0.29	0.42
		0.2340	11.86		-0.09	0.0081					
		0.2350	11.92		-0.03	0.0009					
6	14	0.2760	14.07	13.95	0.12	0.0144	0.102	0.73	0.253	13.95±0.25	0.36
		0.2724	13.88		-0.07	0.0049					
		0.2728	13.91		-0.04	0.0016					
7	17.5	0.3410	17.49	17.39	0.10	0.0100	0.105	0.60	0.261	17.39±0.26	0.63
		0.3370	17.28		-0.11	0.0121					
		0.3390	17.39		0.00	0.0000					

$$b = \frac{n \cdot \Sigma xi \cdot yi - \Sigma xi \cdot \Sigma yi}{n \cdot \Sigma xi^2 - (\Sigma xi)^2} = \frac{7 \cdot 16,572 - 69,5 \cdot 1,366}{7 \cdot 850,25 - 69,5^2} = 0,0188 \approx 0,019; \quad (12)$$

$$Sy^2 = \frac{\sum (Yi - Y_{hisob.})^2}{n-1} = \frac{0,00000291}{7-1} = \frac{29,1 \cdot 10^{-6}}{6} = 4,85 \cdot 10^{-6}; \quad (13)$$

$$Sb = \sqrt{\frac{Sy^2}{\sum Xi^2}} = \sqrt{\frac{4,85 \cdot 10^{-6}}{850,25}} = 7,5 \cdot 10^{-5}; \quad (14)$$

$$\Delta_b = tg\alpha Sb = 9,2 \cdot 7,5 \cdot 10^{-5} = 6,94 \cdot 10^{-4}; \quad (15)$$

$$\overline{b} = b \pm tga \cdot Sb = 0,019 \pm 9,2 \cdot 7,5 \cdot 10^{-5} = 1,9 \cdot 10^{-2} \pm 6,94 \cdot 10^{-4}. \quad (16)$$

The accuracy and precision of the detection method were calculated using the "added–found" method. The value of the relative standard deviation (S_r) did not exceed 1.51%, the value of the deviation from the mean (ΔX) did not exceed 0.290 Table 6, and (Pagliano et al. 2015) the lower limit of determination (Q_{min}) did not exceed 0.247 µg/25 mL Table 7.

$$Q_{min} = \frac{5 \cdot \overline{S}_A \cdot M \cdot V \cdot B \cdot 1000}{\varepsilon_{real} \cdot \ell} = \frac{5 \cdot 0,00155 \cdot 25 \cdot 65,39 \cdot 1000}{51824 \cdot 1} = 0,247\ \mu g/25\ mL \quad (17)$$

Table 7 Results of determining Q_{min} of Zn(II) using the BHNDS-2.7 reagent (n = 3, P = 0.95)

No.	Zn(II) added. μg	$A_{1.2.3}$	$\bar{A}$	Found Zn(II). μg	$(A_i–\bar{A})$	$(A–\bar{A})^2 \cdot 10^{-6}$	$S_A \cdot 10^{-3}$	$\overline{S}_A$
1	2.0	0.0464	0.0463	1.99	0.0001	0.010	0.50	0.00155
		0.0468		2.02	0.0005	0.250		
		0.0458		1.96	-0.0005	0.250		
2	6.0	0.1230	0.1224	6.02	0.0006	0.360	0.57	
		0.1222		5.98	-0.0002	0.040		
		0.1219		5.96	-0.0005	0.250		
3	8.0	0.1620	0.1604	8.07	0.0016	2.560	1.70	
		0.1605		7.99	0.0001	0.010		
		0.1586		7.89	-0.0018	3.240		
4	10.0	0.2000	0.1980	10.07	0.0020	4.000	2.00	
		0.1980		9.97	0.0000	0.000		
		0.1960		9.86	-0.0020	4.000		
5	12.0	0.2380	0.2357	12.07	0.0023	5.290	2.08	
		0.2340		11.86	-0.0017	2.890		
		0.2350		11.92	-0.0007	0.490		
6	14.0	0.2760	0.2737	14.07	0.0023	5.290	1.97	
		0.2724		13.88	-0.0013	1.690		
		0.2728		13.91	-0.0009	0.810		
7	17.5	0.3410	0.3390	17.49	0.0020	4.000	2.00	
		0.3370		17.28	-0.0020	4.000		
		0.3390		17.39	0.0000	0.000		

Table 8 Analytical parameters of Zn(II) determination with BHNDS-2,7

No.	Analytical parameter	Value
1	Maximum absorption wavelength, λ (nm)	580
2	Optimal pH	5.3
3	Medium	Universal buffer
4	Stability time of the complex	8 h
5	Reagent volume, mL	1.2
6	Linear range (Beer–Lambert law), μg/mL	1.0–17.5
7	Bathochromic shift, Δλ (nm)	85
8	Sensitivity (Sandell's sensitivity), μg/cm^2	0.001
9	Composition of the complex (M:R)	1:2
10	Molar absorptivity, ε (L mol^{-1} cm^{-1})	51,282
11	Equilibrium constant, Keq	0.03
12	Stability constant, lgβ (ZnR_2)	18.96
13	Dissociation constant of the reagent, K_{diss}	$6.71 \cdot 10^{-8}$
14	Molar absorptivity of the reagent, ε	2704
15	Slope (b)	0.019
16	Intercept (a)	0.0086
17	Coefficient of determination (R^2)	0.9995
18	Correlation coefficient (r)	0.9997
19	Relative standard deviation, Sr (%)	1.51
20	Confidence interval, ΔX	0.290
21	Limit of detection (Q_{min}), μg/25 cm^3	0.247

Here: ε_{real} is the actual molar absorptivity, V is the volume of the solution, B is the atomic mass of the element, ℓ is the optical path length of the cuvette, M is the number of number of Zn(II) atoms in the complex, S_A is the standard deviation, and Q_{min} is the lower limit of detection.

The following analytical parameters were determined during the study Table 8:

The influence of interfering ions on the spectrophotometric determination of Zn(II) (15 μg) with the BHNDS-2,7 reagent was investigated Table 9.

The effect of interfering ions on the spectrophotometric determination of the Zn(II) ion (15.0 μg) was studied using the BHNDS-2.7 reagent. It was found that the following ions do not interfere with the detection of Zn ions in the given ratio: Na^+, K^+, Ba^{2+}, Mg^{2+}, Cl^-, Br^-, F^-, J^-, NO_3^-, $S_2O_3^{2-}$, CH_3COO^-, $C_2O_4^{2-}$, PO_4^{3-}, $Cr_2O_7^{2-}$, ClO_4^- (1:1000); Al^{3+}, Pb^{2+}, NO_2^-, SO_4^{2-}, SiO_3^{2-} (1:100); Sn^{2+}, (1:10); Zn(II) (1:7,5); Bi^{3+}, Ni(II), Cr^{3+} (1:5), Mn^{2+}, Hg^{2+}(1:1). The interfering ions are: Co^{2+} (1:1), Cd^{2+}, Fe^{2+}, SCN^-, (1:0,5), EDTA, CN^- (1:0,1). It is eliminated by masking the interfering ions, i.e., Fe^{2+} ions are initially oxidized to Fe^{3+} by H_2O_2, and Co^{2+}, Fe^{3+}, and Cd^{2+} ions can be masked by SCN^-, EDTA, and CN^-. If the amount of metal ions is high, EDTA can be masked, followed by EDTA masking with Ca^{2+} ions. The application of appropriate masking agents significantly reduced the influence of interfering ions and improved the selectivity of the proposed method.

To verify the accuracy and precision of the method, it was applied to an artificial mixture using the ''added-found''

Table 9 Effect of interfering cations and anions on the determination of Zn(II)

No.	Interfering ion	Mass ratio (Zn(II):Ion$^{n\pm}$)	Found Zn(II). µg	Relative error (%)	Interference
1	Na^+	1:1000	14.68	2.13	-
2	K^+	1:1000	15.15	1.0	-
3	Ba^{2+}	1:1000	14.54	3.07	-
4	Cd^{2+}	1:0.5	12.38	17.47	Interferes
5	Zn(II)	1:7.5	14.12	4.93	-
6	Fe^{2+}	1:0.5	14.10	6.00	Interferes
7	Al^{3+}	1:100	14.65	2.33	-
8	Bi^{3+}	1:5	14.44	3.73	-
9	Mn^{2+}	1:1	14.64	2.4	-
10	Hg^{2+}	1:1	14.46	3.6	-
11	Cr^{3+}	1:5	14.29	4.73	-
12	Co^{2+}	1:1	11.78	21.47	Interferes
13	Ni(II)	1:5	14.84	1.07	-
14	Mg^{2+}	1:1000	14.49	3.4	-
15	Sn^{2+}	1:10	14.32	4.53	-
16	Pb^{2+}	1:100	15.36	2.40	-
17	J^-	1:1000	15.24	1.60	-
18	Cl^-	1:1000	15.34	2.27	-
19	Br^-	1:1000	14.85	1.0	-
20	F^-	1:1000	14.85	1.0	-
21	NO_3^-	1:1000	14.63	2.47	-
22	$S_2O_3^-$	1:1000	15.38	2.53	-
23	CH_3COO^-	1:1000	15.47	3.13	-
24	SCN^-	1:0.5	10.53	29.80	Interferes
25	CN^-	1:0.1	11.14	25.73	Interferes
26	$C_2O_4^{2-}$	1:1000	14.71	1.93	-
27	SiO_3^{2-}	1:100	14.63	2.47	-
28	PO_4^{3-}	1:1000	15.54	3.60	-
29	$Cr_2O_7^{2-}$	1:1000	14.69	2.07	-
30	NO_2^-	1:100	15.52	3.47	-
31	SO_4^{2-}	1:100	15.62	4.13	-
32	ClO_4^-	1:1000	14.66	2.27	-
33	EDTA	1:0.1	11.46	23.60	Interferes

method Table 10. Cl^-, NO_2^- ions were added in a 1:60 ratio to mask foreign ions that interfere with the detection of Zn (II) ions. Based on the results obtained, it is possible to identify Zn (II) in natural objects of similar composition. At the same time, the relative Sr≤0.021 did not exceed.

Application for real samples

The developed method was successfully applied to the analysis of ore samples from the Khandiza deposit (Surkhandarya region, Uzbekistan), enabling the simultaneous determination of Zn(II) and associated elements.

Ore composition, %: Zn-12.37; Cu-11.45; Au-0.001; Ag-0.00131; Mn-0.6; Cd-0.1; Pb-3.32; Fe_2O_3-26.42; S-22.4; SiO_2-52.67.

Sample preparation for analysis: A 0.1000 g ore sample was decomposed according to the recommended procedure. The sample was placed in a heat-resistant beaker and heated until complete dissolution. After cooling, the solution was diluted with bidistilled water and filtered. The residue was washed three times with 5 mL portions of bidistilled water, and the washings were combined with the filtrate. The residue remaining on the filter was transferred to a separate beaker and treated with 6–8 mL of 5 M HCl to ensure complete dissolution of Zn-containing compounds. After filtration, the resulting solution was quantitatively transferred into a volumetric flask and diluted to the mark with bidistilled water. The prepared solution was subsequently used for spectrophotometric determination of Zn(II) (Merlo et al. 2023).

Determination method: a volumetric flask of 25 mL was filled with 1.2 mL of a 0.05% BHNDS-2.7 reagent solution, 5 mL of a universal buffer solution with a pH of 5.3, an appropriate volume of the sample solution, and a masking agent at calculated concentrations using 10.0 mL of F^- to mask Fe^{3+}, then the flasks were diluted with bidistillate water to the mark. The optical densities of the solutions were measured on an EMC-31PC-UV spectrophotometer at wavelengths of λ = 580 nm relative to the reference solution.

As can be seen from the results presented in Table 11, the amount of Zn (II) found during the spectrophotometric determination of the ore composition of the Khandiza deposit with the BHNDS-2,7 Zn(II) ion is in good agreement with the expected valueof Zn (II), where Sr≤1.66% was not exceeded. The developed spectrophotometric method allows for the determination of Zn (II) ions in ore samples with the aforementioned composition.

Table 10 Results of Zn(II) determination in an artificial mixture

Mixture composition µg	Zn(II) added. µg	**A**	Found Zn(II). µg X_i	S	S_r%	X±ΔX
K^+. Na^+ (600); Bi^{3+}. Co^{2+} (0.2);NO_2^-. Cl^- (60); Hg^{2+} (0.02); Ba^{2+}. NO_3^- (800); CH_3COO^-(200);	12.0	0.2370	12.01	0.246	2.00	12.12±0.31
		0.2440	12.38			
		0.2400	12.17			
		0.2320	11.75			
		0.2420	12.27			

Table 11 Results of spectrophotometric determination of Zn(II) in ore samples from the Khandiza deposit (m_{sample} = 0.1000 g, n = 3, P = 0.95)

No.	V_{sample}. ml	Zn(II) in sample. µg	$A_{1.2.3}$	Found Zn(II). µg	$\overline{X}_i \pm \Delta X$	$X_i - \overline{X}$	$(X_i - \overline{X})^2$ S		Sr%
λ = 580 nm. pH = 5.3									
1	0.25	3.09	0.0650	2.97	3.02 ± 0.124	-0.05	0.0025	0.05	1.66
			0.0660	3.02		0.00	0.0000		
			0.0670	3.07		0.05	0.0025		
2	0.50	6.18	0.1250	6.13	6.11 ± 0.203	0.02	0.0004	0.0819	1.34
			0.1260	6.18		0.07	0.0049		
			0.1230	6.02		-0.09	0.0081		
3	1.00	12.37	0.2440	12.39	12.27 ± 0.273	0.12	0.0144	0.1098	0.89
			0.2410	12.23		-0.04	0.0016		
			0.2400	12.18		-0.09	0.0081		

Table 12 Comparison of optimal conditions and analytical characteristics of the proposed method for Zn(II)

No.	Reagent	pH	Zn:R molar ratio	Beer law range (µg/25 mL)	ε (L mol^{-1} cm^{-1})	References
1	BHNDS-2,7	5,3	1:2	1.0–17.5	51 282	This work
2	N- (2- (2-amino-5-nitrophenyl) diazenyl) ethyl) naphthalen-1-amine	11.0	1:1	0.653 – 6.538	8740	Abduljaleel et al. (2025)
3	murexide	7.0	1:2	5–50.0	19 500	Muftah Elsherif et al. (2022)
4	4-((4,5-diphenyl-imidazol-2-yl) diazenyl)-3-hydroxynaphthalene-1 -sulfonate	10.5	1:2	1.0–22.0	2229	Ameer and Hussain (2023)
5	2-[(2-methylphenyl)azo]-4,5-diphenyl imidazol	6.5	1:1	1.0–50.0	5994	Jawad and Mohummed Husien (2018)
6	(E)-2-(4,5-bis(4-methoxyphenyl)-1H-imidazole-4-yl) diazenyl) benzoic acid	6.8	1:2	0.5–15	3717	Radhi (2024)

Table 13 Assessment of the competitiveness of the proposed spectrophotometric method for Zn(II) determination

No.	Analytical methods	Buffer solution (pH)	Q_{min}, µg/mL	Linear range (µg/25 mL)	S_r	ε (L mol^{1} cm^{-1})	References
1	Proposed method	Universal buffer (pH 5.3)	0.0099	1–17.5	1.51	51 282	This work
2	Liquid ion exchange method	Acetate–acetic acid buffer (pH 10)	0.839	0.1–10	3.1	5422	Hameed et al. (2024)
3	Cloud point extraction	Universal buffer (pH 9.0)	0.01	0.01–0.8	4	7342	Preparations et al. (2023)

Comparison of the proposed method with other reagents and reported analytical methods

The developed method was compared with previously reported spectrophotometric methods based on other analytical organic reagents Table 12, as well as with alternative analytical methods Table 13. The comparison results indicate that the proposed method is superior or at least comparable to existing methods in terms of sensitivity, selectivity, and accuracy. The high sensitivity of the proposed method can be attributed to the high molar absorptivity coefficient (ε = 51,282 L mol^{-1} cm^{-1}), the high stability constant of the Zn(II)–BHNDS-2,7 complex (logβ = 18.96), and the pronounced bathochromic shift (Δλ = 85 nm), which enhances spectral contrast. These factors collectively contribute to the low detection limit and reliable determination of trace amounts of Zn(II).

Conclusions

A sensitive and selective spectrophotometric method for the determination of Zn(II) based on the BHNDS-2,7 reagent is proposed. Optimal conditions (λ = 580 nm, pH = 5.3, in a universal buffer medium) were determined, and the formation of the complex with a composition of Zn: BHNDS-2,7 = 1:2 was confirmed by various physicochemical methods. Based on UV–Vis, IR, and ^{1}H NMR spectroscopic data, the structure of the Zn(II)–BHNDS-2,7 complex was proposed. The proposed method has a high molar absorption coefficient (ε = 51,282 L mol^{-1} cm^{-1}), a wide linearity range (1.0–17.5 µg/25 mL), a low detection limit (Q_{min} = 0.247 µg/25 mL), and high accuracy (R^2 = 0.9995; Sr ≤ 1.66%). The influence of extraneous ions was investigated, and the possibility of effective masking of interfering ions was shown. The developed method was successfully

applied in ore samples of the Khandiza deposit, and a high correspondence of the experimental results was established. The obtained results confirm the reliability, sensitivity, and practical effectiveness of the method for determining Zn (II) ions in real objects of complex composition. The proposed method shows comparable or superior sensitivity, selectivity, and precision versus existing reagents and techniques, confirming its competitiveness for Zn(II) determination. The high sensitivity, selectivity, and stability of the Zn(II)–BHNDS-2,7 system demonstrate its suitability for trace Zn(II) determination in complex samples. The proposed method combines simplicity, favorable analytical performance, and practical applicability, making it a competitive alternative for environmental, industrial, and geological analyses.

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Data availability No datasets were generated or analysed during the current study.

Declarations

Competing interests The authors declare no competing interests.

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