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Coordination Chemistry of Zinc(II) Dithiocarbamate with Polypyridine Ligands: Synthesis and Detailed Spectroscopic Insights

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KEYWORDS

Zinc(II) dithiocarbamate; 1,10-phenanthroline; 2,2′-bipyridine; coordination complexes; FT-IR and NMR spectroscopy; UV–visible spectroscopy.

ABSTRACT:

Zinc(II) dithiocarbamate complexes continue to attract considerable attention due to their versatile coordination behaviour, rich spectroscopic features, and relevance in coordination and materials chemistry. In the present study, a parent zinc(II) dithiocarbamate complex, bis{[4-(piperidin-1-yl)piperidine]carbodithioato-κ²S,S′}zinc(II), and its coordination adducts with the neutral nitrogen-donor ligands 1,10-phenanthroline and 2,2′-bipyridine were successfully synthesized. The complexes were obtained in good yields and characterized comprehensively using elemental analysis, ultraviolet–visible (UV–Vis) spectroscopy, Fourier-transform infrared (FT-IR) spectroscopy, and nuclear magnetic resonance (^{1}H and ^{13}C NMR) spectroscopy. Elemental analysis confirmed the proposed stoichiometry and high purity of the synthesized compounds. FT-IR spectra revealed characteristic thioureide ν(C–N) and ν(C–S) bands, supporting bidentate coordination of the dithiocarbamate ligand through sulphur atoms, while additional ν(Zn–N) bands in the adducts confirmed coordination of the nitrogen-donor ligands. Electronic absorption spectra were dominated by ligand-centred and charge-transfer transitions, consistent with the d^{10} electronic configuration of Zn(II). The ^{1}H and ^{13}C NMR spectra provided definitive evidence for dithiocarbamate formation, retention of the $Zn(dtc)_2$ core, and ligand-induced electronic modulation, particularly through systematic downfield shifts of the thioureide carbon resonance. Overall, the combined spectroscopic results clearly establish the coordination environment and electronic features of the parent and adduct complexes, highlighting the influence of polypyridine ligands on the structural and spectroscopic properties of zinc(II) dithiocarbamate systems.

1.INTRODUCTION

Dithiocarbamate ligands are a versatile and widely studied class of organosulfur ligands, because of their high chelating ability towards a wide range of metal ions and their ability to create structurally varied coordination complexes[1].Due to their special electronic and structural characteristics, these ligands have been used in materials chemistry, biological systems, and catalysis. They usually coordinate through their two sulphur atoms, stabilising transition and post-transition metal centres[2].Zinc(II) dithiocarbamate complexes, in particular, have garnered considerable interest due to the d^{10} electronic configuration of Zn^{2+}, which often leads to flexible coordination geometries and rich spectroscopic behaviour[3]. In coordination chemistry, these complexes offer useful platforms for investigating structure–property correlations.Prior research has shown that interactions with neutral nitrogen-donor ligands can alter the coordination sphere of zinc dithiocarbamates, producing mixed-ligand

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complexes with unique structural and electrical properties[4].

Bidentate nitrogen donors known as polypyridine ligands, such as 1,10-phenanthroline and 2,2′-bipyridine, easily form stable chelates with metal centres. The binding environment, electronic absorption characteristics, and general shape of the resultant assemblies are frequently impacted by their integration into metal complexes[5].These ligands are perfect for research combining spectral, structural, and electronic analysis because they are known to improve coordination complex stability and contribute to distinctive spectroscopic signatures[6].

In this work, we describe the synthesis of a parent complex of zinc dithiocarbamate and its coordination adducts with 2,2′-bipyridine and 1,10-phenanthroline. Elemental analysis, Fourier-transform infrared (FT-IR) spectroscopy, ultraviolet–visible (UV-Vis) spectroscopy, and nuclear magnetic resonance (^{1}H and ^{13}C NMR) spectroscopy were used to thoroughly characterise the resultant complexes. These methods enable a deeper comprehension of the coordination behaviour and spectroscopic characteristics intrinsic to this class of zinc complexes by offering comprehensive insights into the molecular composition, coordination environment, ligand field effects, and structural alterations linked to adduct formation[7].

2.EXPERIMENTAL

All reagents and solvents employed in the present study were of analytical grade and were used as received without any further purification (Sd Fine Chemicals). Infrared spectra were recorded in the 400–4000 cm^{-1} region using an FT-IR spectrometer, while electronic absorption spectra were measured at room temperature on a UV–visible spectrophotometer. NMR measurements were carried out on a 400 MHz spectrometer using standard procedures. Elemental analysis (CHNS) was performed by instrumentation, and Zn content was determined from gravimetric analysis using anthranilic acid. All instrumental techniques were operated according to standard protocols to ensure accuracy and reproducibility of the spectral and structural data.

2.2. Synthesis of Complexes

2.2.1.Preparation of bis{[4-(piperidin-1-yl)piperidine]carbodithioato-κ²S,S'}zinc(II);

[Zn(4-pip$_2$dtc)$_2$] (1)

4-Piperidinopiperidine (0.4 g, 2 mmol) and carbon disulfide (1 ml, 2 mmol) were dissolved in ethanol (10 ml) under ice-cold conditions (5 °C) with constant stirring to form a white solution of the corresponding dithiocarbamic acid. To this solution, an aqueous solution of zinc(II) chloride (0.172 g, 1 mmol) was added dropwise under continuous stirring. The reaction mixture immediately yielded pale yellowish precipitate, which was collected by filtration, washed thoroughly with ethanol, and dried. The complex was obtained in good yield, 74%.

2.2.2.Preparation of (1,10-phenanthroline-N,N′)bis(4-(piperidin-1-yl)piperidine-1-carbodithioato-S,S')zinc(II)

A hot ethanolic solution of 1,10-phenanthroline (0.09g,0.5mml) was added to a hot ethanolic solution (1:1) of [Zn(4-pip$_2$dtc)$_2$] , (0.19g,

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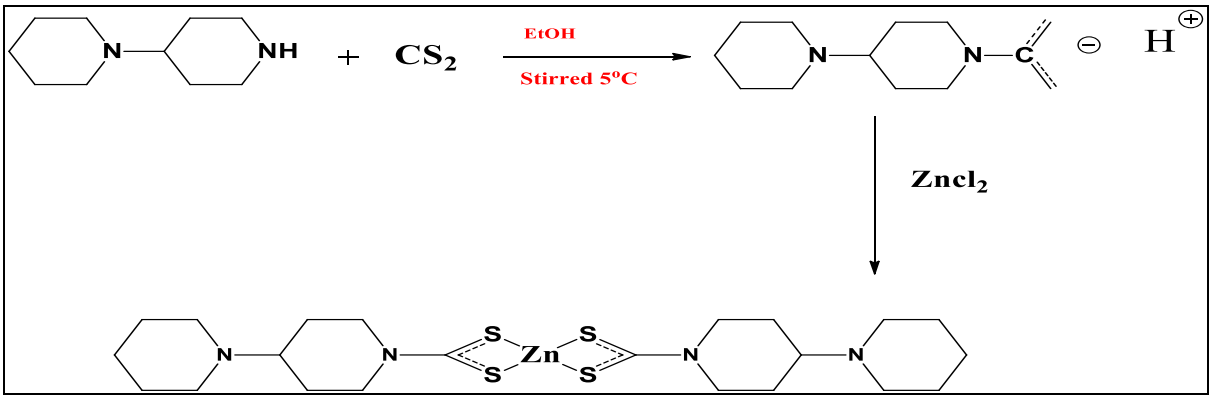
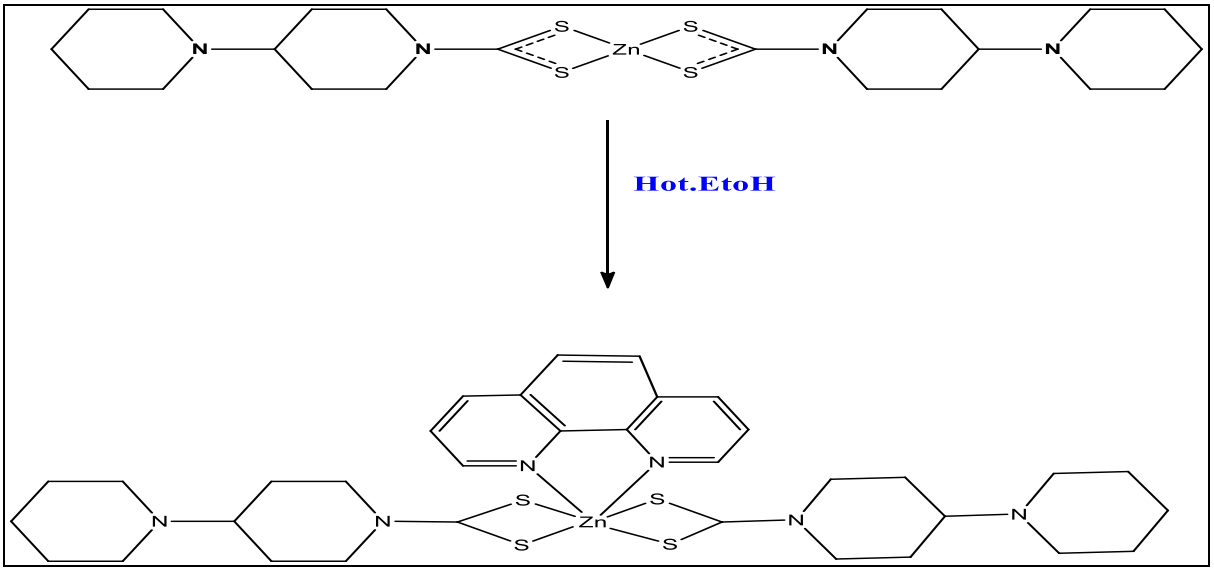
0.5mmol).The resulting yellow mixture was heated for few minutes and was left for evaporation at room temperature, and the resulting complex was precipitated, collected by filtration, and washed thoroughly with ethanol. The complex was dried and obtained in good yield, 70%.

2.2.3. Preparation of (2,2′-bipyridine-N,N′)bis(4-piperidin-1-yl)piperidine-1-carbodithioato- S,S')zinc(II)

A hot solution of 2,2′-bipyridine (0.078g,0.5mmol) in ethanol was mixed with hot solution of [Zn(4-pip$_2$dtc)$_2$] , (0.19g,0.5mmol) in ethanol. The resulting yellow mixture was heated for few minutes and was left for evaporation at room temperature, and the resulting complex was precipitated, collected by filtration, and washed thoroughly with ethanol. The complex was dried and obtained in good yield, 64%.



Scheme 1. Synthetic scheme of complex 1



Scheme 2. Synthetic scheme of complex 2

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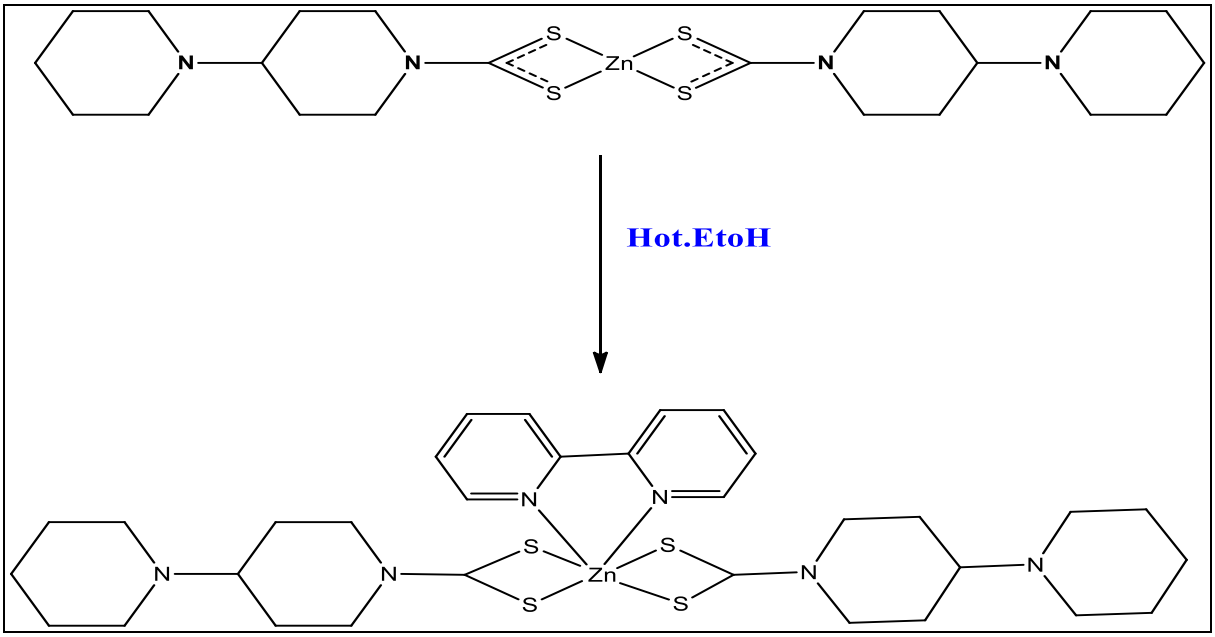

Scheme 3. Synthetic scheme of complex 3

3.RESULT AND DISCUSSION

3.1. Elemental Analysis

Elemental analysis was carried out to verify the molecular formulas and purity of the synthesized zinc dithiocarbamate parent complex and its nitrogen-donor adducts with 1,10-phenanthroline and 2,2-bipyridine. Elemental composition was determined for zinc (Zn), carbon (C), hydrogen (H), nitrogen (N), and sulfur (S) and compared with their corresponding calculated values for the proposed formulas. Elemental analysis data for the complexes showed close agreement with calculated values, supporting the assigned stoichiometries and indicating successful synthesis of the target compounds[8].

For the zinc dithiocarbamate parent complex ($C_{22}H_{40}N_4S_4Zn$), the experimental Zn content (11.8%) closely matched the calculated value (11.79%), confirming the presence of one zinc ion per formula unit. The experimentally determined percentages of carbon (47.24–47.66%), hydrogen (7.01–7.27%), nitrogen (10.02–10.11%), and sulphur (23.01–23.13%) closely matched the corresponding estimated values, demonstrating high sample purity and proper synthesis of the dithiocarbamate ligand environment.

The measured Zn percentage (8.15–8.90%) in the zinc dithiocarbamate-1,10-phenanthroline complex ($C_{34}H_{48}N_6S_4Zn$) matched the theoretical value, indicating that one equivalent of 1,10-phenanthroline was coordinated to the zinc centre. The additional aromatic ligand structure of 1,10-phenanthroline is reflected in the increases in carbon (55.19–55.57%) and nitrogen (11.21–11.44%) compared to the parent complex. Results for sulphur (17.13–17.45%) and hydrogen (6.24–6.59%) matched computed values, confirming the suggested composition and purity.

The measured Zn content (9.20–9.21%) for the zinc dithiocarbamate-2,2-bipyridine complex ($C_{32}H_{48}N_6S_4Zn$) agreed with the expected

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proportion, indicating that one 2,2-bipyridine ligand was incorporated per zinc centre. The percentages of carbon (54.00–54.09%), hydrogen (6.32–6.81%), nitrogen (11.39–11.83%), and sulphur (18.00–18.05%) were similarly in good accord with theoretical values, confirming the expected ligand composition and low levels of contaminants.

While carbon, hydrogen, nitrogen, and sulphur analyses were acquired using a CHNS analyser, zinc was determined in the lab using a conventional wet chemical approach. The effective synthesis, accurate stoichiometry, ligand incorporation, and high purity of the zinc dithiocarbamate parent and its 1,10-phenanthroline and 2,2-bipyridine adducts are confirmed by the strong agreement between estimated and observed values for all elements across all three complexes.

Table 1. Analytical data for the Zn(II) Complexes

Compound	Zn% Exp-Cal	C% Exp-Cal	H% Exp-Cal	N% Exp-Cal	S% Exp-Cal	Melting Point °C
$C_{22}H_{40}N_4S_4Zn$	11.8-11.79	47.24-47.66	7.01-7.27	10.02-10.11	23.01-23.13	170-220
$C_{34}H_4N_6S_4Zn$	8.15-8.90	55.19-55.57	6.24-6.59	11.21-11.44	17.13-17.45	180-240
$C_{32}H_{48}N_6S_4Zn$	9.2-9.21	54.00-54.09	6.32-6.81	11.39-11.83	18.00-18.05	150-200

Table 2. Electronic and FT-IR Data (Cm^{-1})

Complex	**Key λmax (nm)**	**ν_{C-N}**	**ν_{C-S}**	**νzn-s**	**νc-H**	**ν N-H**	**νzn-N**
1.$[Zn(dtc)_2]$	230,270,320	1416	999	439	2936	3432	---
2.$[Zn(dtc)_2(phen)]$	228,258,292,322	1508	1019	419	2925	3423	623
3.$[Zn(dtc)_2(bpy)]$	232,252,285,315	1454	993	406	3053	3431	620

3.2.IR Spectral Studies

The most significant FT-IR absorption bands of the parent Zn(II) dithiocarbamate complex and its mixed-ligand derivatives with 1,10-phenanthroline and 2,2′-bipyridine are summarised in Table 2. The most useful characteristic for evaluating coordination and electrical effects in dithiocarbamate systems is the thioureide ν(C–N) band [9]. The parent ZnDTC complex displays the thioureide ν(C–N) band at 1416 cm^{-1}, which is indicative of partial double-bond character resulting from electron delocalisation inside the $–NCS_2$ moiety, as Table 2 indicates. This band moves

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to higher wavenumbers upon coordination with nitrogen-donor ligands, showing up at 1508 cm^{-1} for the ZnDTC–1,10-phenanthroline complex and 1454 cm^{-1} for the ZnDTC–2,2′-bipyridine complex . This upward change, which results from the redistribution of electron density upon mixed-ligand coordination, unmistakably shows an increase in the thioureide nature of the C–N bond[10]. For metal dithiocarbamate complexes, these shifts in the thioureide band are well-documented and are regarded as compelling proof of coordination actions [11].

Bidentate coordination of the dithiocarbamate ligand through sulphur atoms is supported by the ν(C–S) bands shown in Table 2, which appear in the range 993–1019 cm^{-1}. Zn–S bond formation is further confirmed by the existence of ν(Zn–S) vibrations in the 406–439 cm^{-1} range. Unlike the parent complex, the mixed-ligand complexes exhibit extra bands at around 620–623 cm^{-1}. These bands are attributed to ν(Zn–N) stretching vibrations, indicating that the nitrogen-donor ligands are coordinated[12].

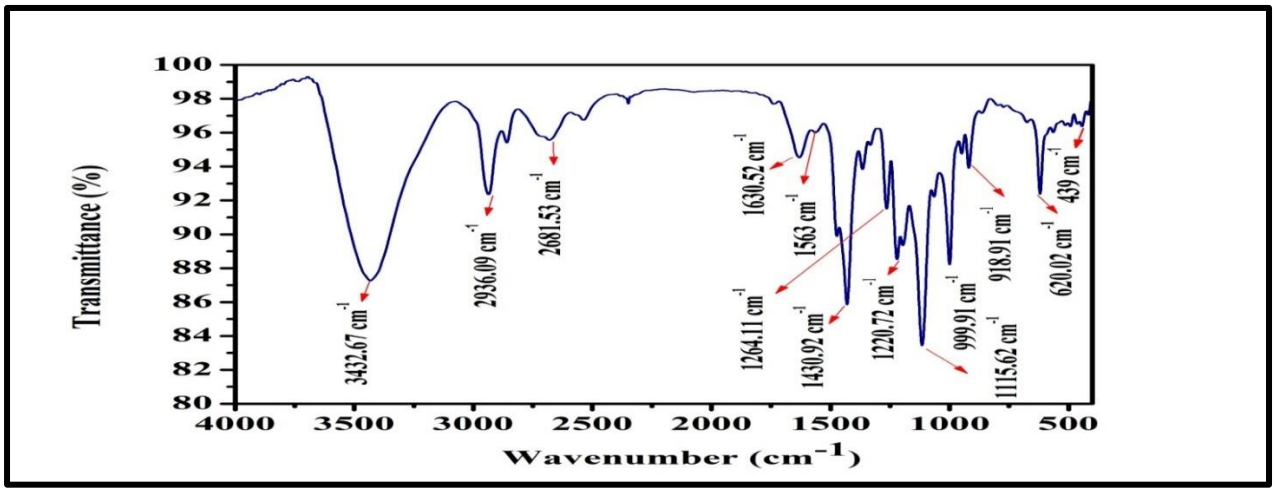
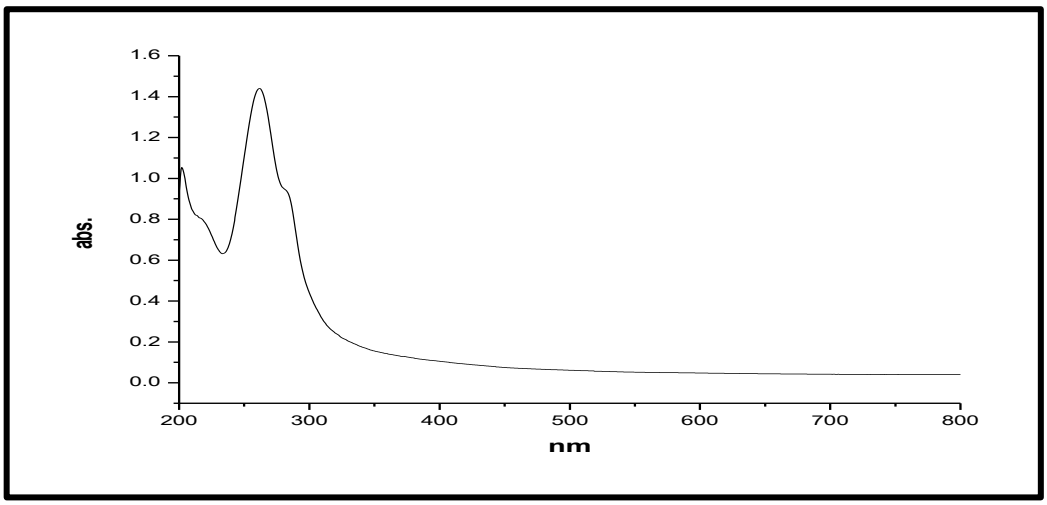


Figure. 1 FT-IR Spectra of Complex 1



(a) UV Spectra of Complex 1

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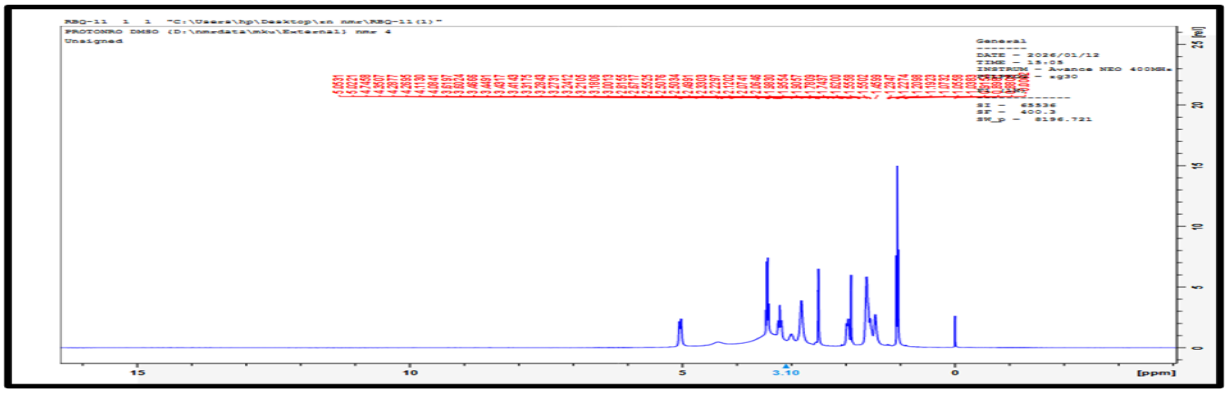
3.3. Electronic Spectral Studies

The electronic absorption spectra of the parent Zn(II) dithiocarbamate complex and its mixed-ligand derivatives with 1,10-phenanthroline and 2,2′-bipyridine (data summarized in Table 2) are dominated by intense absorptions in the ultraviolet region, which is characteristic of d^{10} Zn(II) systems where d–d transitions are absent[13]. The high-energy band in the ~220–240 nm area of the parent ZnDTC complex's spectra is attributed to π → π* intraligand transitions that originate from the conjugated C–N and C=S chromophoric framework of the dithiocarbamate ligand. The n → π* transitions involving the non-bonding electrons of the sulphur atoms in the thiocarbonyl group are responsible for the band that appears in the ~260–280 nm area. The high polarisability and strong donor ability of sulphur atoms cause ligand-to-metal charge transfer (LMCT) transitions from sulphur donor atoms to the Zn(II) centre, which are characterised by a weak and broad absorption tail extending towards ~300–320 nm[14].The mixed-ligand complexes show additional intense absorptions in the ~250–265 nm region upon coordination with nitrogen-donor ligands. These absorptions are attributed to π → π* transitions localised on the aromatic heterocyclic rings of 1,10-phenanthroline and 2,2′-bipyridine, confirming their coordination to the Zn(II) ion. Modest changes in the dithiocarbamate ligand's n → π* transitions are indicative of electronic redistribution brought on by metal–ligand interactions[15].

Because of greater electron removal by the coordinated N-donor ligands, the LMCT band becomes more prominent in the mixed-ligand complexes, especially for the phenanthroline derivative, indicating accelerated sulfur-to-metal charge transfer[16]. The identification of ligand-centered and charge-transfer transitions as the predominant electronic processes is supported by the lack of absorption bands in the visible range for all complexes, which further validates Zn(II)'s closed-shell d^{10} electronic structure[17].

In addition, the absence of ligand-field-dependent d–d transitions and the dominance of ligand-centered and charge-transfer bands are consistent with a tetrahedral or distorted tetrahedral coordination environment around the Zn(II) centre, as expected for d^{10} metal complexes where electronic spectra are largely insensitive to geometry[18].

^{1}H NMR Spectra of Complex 1



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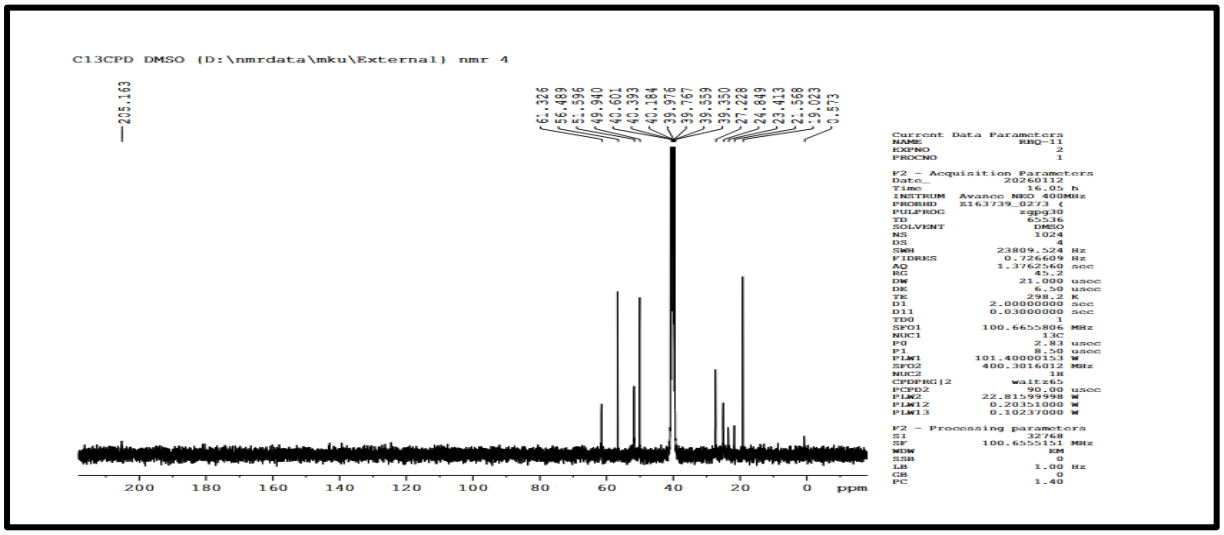

^{13}C NMR Sectra of Complex 1

3.4 ^{1}H NMR Spectral Analysis

The ^{1}H NMR spectrum of the parent Zn(II) dithiocarbamate complex, $Zn(dtc)_2$, displays characteristic resonances corresponding to the piperidinopiperidine framework, confirming successful dithiocarbamate formation . The aliphatic CH_2 protons of the piperidine rings are identified as multiplets in the range δ 1.2–2.5 ppm, whereas signals at δ 2.8–3.6 ppm correspond to N–CH_2 protons, which are shifted downfield relative to the free amine because of electron withdrawal linked to dithiocarbamate formation and Zn(II) coordination [19]. The N–H proton is responsible for a large resonance at δ 4.8–5.1 ppm, which shows that the secondary amine proton is unaltered during complexation. This characteristic is frequently noted for zinc dithiocarbamate systems[20]. Sharp and well-resolved signals without paramagnetic widening are produced by Zn(II)'s diamagnetic d^{10} electronic structure. The spectra of complexes 12–14 show clear alterations upon coordination with neutral donor ligands. In complex 13, coordinated 1,10-phenanthroline is responsible for novel aromatic resonances in the region δ 7.2–9.2 ppm. The most downfield signals are attributed to H_2/H_4 protons next to the coordinating nitrogen atoms, which is consistent with chelation to Zn(II) [21]. Similarly, coordinated 2,2′-bipyridine causes complex 14 to show aromatic signals in the range δ 8.0–8.8 ppm, with discernible downfield changes in relation to the free ligand, indicating Zn–N bond formation [22]. The integrity of the $Zn(dtc)_2$ core, successful coordination of neutral ligands, and high purity of the synthesised compounds are all confirmed in all complexes by the retention of the aliphatic piperidine resonances, the appearance of ligand-specific aromatic signals, and the absence of extraneous peaks [23].These findings are entirely in line with the ^{1}H NMR properties of coordination derivatives of zinc dithiocarbamate complexes that have been described [24] [25].The data is given in **Table 3.**

3.5.^{13}C NMR Spectral Studies

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The ^{13}C NMR spectra of the parent Zn(II) dithiocarbamate complex and its coordination derivatives provide strong evidence for dithiocarbamate formation and ligand-induced electronic effects, with particular diagnostic importance attributed to the thioureide carbon

(–NCS_2) resonance. The thioureide carbon in $Zn(dtc)_2$ appears in the highly deshielded region about 200 ppm, which is indicative of a delocalised C=S group resulting from coordination through sulphur atoms to the Zn(II) center and significant π-electron delocalisation between the C–N and C–S bonds[26] [27] [28]. Systematic downfield shifts of this resonance, which represent variations in electron density within the dithiocarbamate framework, are seen upon coordination with neutral nitrogen-donor ligands. The 1,10-phenanthroline complex shows a downfield shift to $\delta \approx 208$ ppm, which is consistent with higher chelation and σ-donation by the bidentate phenanthroline ligand, which improves metal–nitrogen interaction and lowers electron density at the thiocarbonyl carbon [7] .The 2,2′-bipyridine complex exhibits the greatest deshielding, with the thioureide carbon appearing at $\delta = 209$ ppm, indicating more efficient π-acceptor interactions and heightened competition for electron density at the Zn(II) core[29].Furthermore, resonances between δ ~45–65 ppm and δ ~20–30 ppm correspond to N–CH_2 and aliphatic carbons of the piperidinopiperidine moiety, respectively, and signals in the region δ ~120–150 ppm are assigned to aromatic carbons of the coordinated ligands. The corresponding ^{13}C NMR data for the other synthesised complexes are summarised in **Table 4**. In line with documented zinc dithiocarbamate systems[30]the progressive downfield shift of the thioureide carbon throughout the series verifies retention of the $Zn(dtc)_2$ core and very well illustrates ligand-dependent electronic modulation inside the dithiocarbamate unit.

Table 3.^{1}H NMR spectral data of synthesized compounds

Complex	δ(ppm)	Assignment
$[Zn(dtc)_2]$(Parent)	1.2–2.5	Aliphatic CH_2 (piperidine rings)
	2.8–3.6	N–CH_2
	4.8–5.1	N–H
[(Zn(dtc)$_2$–phen)]	7.2–9.2	Aromatic protons (1,10-phenanthroline)
	8.9–9.2	H_2/H_4 (phen)
[(Zn(dtc)$_2$–bipy)]	8.0–8.8	Aromatic protons (2,2′-bipyridine)
	1.2–3.6	Aliphatic & N–CH_2

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Table 4. ^{13}C NMR spectral data of synthesized compounds

Complex	δ (ppm)	Assignment
[Zn(dtc)$_2$ (Parent)]	205	Thioureide C=S (–NCS$_2$)
	45–65	N–CH$_2$ carbons
	20–30	Aliphatic CH$_2$ carbons
[Zn(dtc)$_2$(1,10phenanthroline)]	208	Thioureide C=S
	120–150	Aromatic carbons (phen)
	45–65	N–CH$_2$ carbons
[Zn(dtc)$_2$–(2,2′-bipyridine)]	209	Thioureide C=S
	120–150	Aromatic carbons (bipy)
	45–65	N–CH$_2$ carbons
	20–30	Aliphatic CH$_2$ carbons

4.CONCLUSION

The parent zinc(II) dithiocarbamate complex and its coordination adducts with 1,10-phenanthroline and 2,2′-bipyridine were successfully synthesized and comprehensively characterized using elemental analysis, UV–visible spectroscopy, FT-IR spectroscopy, and multinuclear NMR techniques. Elemental analysis confirmed the proposed stoichiometries and high purity of all complexes. FT-IR spectra established bidentate coordination of the dithiocarbamate ligand through sulphur atoms and additional Zn–N bond formation in the phenanthroline and bipyridine complexes. Electronic spectra revealed ligand-centered and charge-transfer transitions characteristic of d^{10} Zn(II) systems, with no evidence of d–d transitions.

The Zn(dtc)$_2$ core retention, complex formation, and ligand-dependent electronic modulation were all well supported by the ^{1}H and ^{13}C NMR spectra. Changes in electron density within the dithiocarbamate framework were clearly shown by diagnostic thioureide carbon resonances and regular downfield shifts upon coordination with nitrogen-donor ligands. Overall, this study demonstrates how polypyridine ligands affect the spectroscopic and structural characteristics of zinc dithiocarbamate complexes and validates their applicability as well-defined model systems for examining structure–property connections in zinc coordination chemistry.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal

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relationships that could have appeared to influence the work reported in this paper.

Data availability

Data will be available upon request.

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