

SYNTHESIS OF SUBSTITUTED 2-AMINO OXAZOLES WITH THEIR COBALT(II) AND PLATINUM(IV) COMPLEXES AND EVALUATION OF THEIR BIOLOGICAL ACTIVITY

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ABSTRACT. In this research we successfully used substituted acetophenone or acetyl furan with urea in existence of iodine to synthesis novel substituted 2-amino oxazoles (1-3) from substituted acetophenone or acetyl furan with urea in presence of iodine. The structures of (1-3) were proved by spectroscopic methods (IR, ¹H NMR, ¹³C NMR, and C.H.N.). Compounds (1-3) were used in 2:1 ratio to synthesis of three complexes by Reaction with (CoCl₂.6H₂O) (4-6), and three complexes by reaction with (PtCl₄) (7-9) respectively in the presence of triethylamine in absolute ethanol. The thermal analysis (TGA), magnetic characteristics, and differential thermal analysis (DTA) was studied. Number of manufactured compounds were assessed versus the growth of four kinds of bacteria.

KEY WORDS: Oxazole, cobalt complex, platinum complex, TGA, DTA

INTRODUCTION

Oxazoles are one of the most widespread heterocyclic compounds. They are biologically active molecules and natural products that have received interest in both industry and academic research. In particular, the 2,5-substituted or 2,4,5-substituted oxazoles are found in many natural products are pharmacologically active molecules, including antibacterial and anti-pancreatic cancer agents and antithrombotic such as ditazole Figure 1 [1-3].

Numerous approaches have been described for synthesizing 2-aminooxazoles. These include reacting a terminal alkyne with a nitrile in the presence of a gold catalyst, combining an isocyanide with thionyl chloride and an aldehyde, and the reaction between an α -bromoketone and urea [4].

Inorganic chemistry research in the medical field has been widely developed through the preparation of new complexes that are believed to have better biological activity than asymmetric compounds [5]. The complexes consist of metal ions, which are metals that contain d-orbitals that are not filled with electrons, with ligands that have electron-donating atoms. Complexes containing cobalt have better antibacterial activity than nickel and copper complexes [6].

Platinum complexes are one of the most important chemotherapy treatments used in the treatment of cancer [7, 8]. Cisplatin (cis-diamine dichloroplatinII) or oxaliplatin and carboplatin binds directly inside cancer cells to the DNA bases[9], which leads to their bonding with its molecules, prevents cell division and leads to apoptosis. For cells, the use of the effective dose of these drugs causes toxicity and significant side effects, and the toxicity of these drugs raises interest in scientific research for other types of platinum complexes due to the possibility of their great effectiveness against cancer diseases [10]

In this study, oxazole compounds (1-3) were prepared and categorized by micro elemental analysis (C.H.N.) and TGA plus spectroscopic techniques (UV-Vis, FTIR, ¹H-NMR, and mass). Atomic absorption, elemental analysis, infrared, GC-Mass, TGA, and UV-Vis spectral methods, as well as conductivity and magnetic susceptibility, were used to characterize the complexes of Co(II) (4-6), and Pt(IV) (7-9). These compounds were used to prepare cobalt complexes and platinum complexes. These complexes can be used in cancer treatment and as catalysts.

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EXPERIMENTAL

Materials and methods

All chemicals and solvents were purchased from commercial suppliers additional purification. We verified the IR spectra (ν_{\max} in cm^{-1}) using a Bruker FT-IR 8400 spectrophotometer with KBr discs. Additionally, $^1\text{H-NMR}$ spectroscopy was performed on a Bruker instrument operating at 400 MHz, employing DMSO- d_6 as the solvent and TMS as a standard for chemical shift referencing. The molar conductivity measurement was carried out using a Conductivity Meter-Model (Eutech pc700) device, The magnetic susceptibility of the prepared complexes was measured using a Magnetic Susceptibility Balance device equipped by Sherwood Scientific Company, Ultraviolet and visible measurements of the ligand and complexes were recorded using a UV Spectrophotometer PG INSTRUMENTS device .

Preparation of 2-Amino oxazoles compounds

A mixture of 0.02 (substituted acetophenone or 2-acetylfuran) with (0.02 mol, 1.2 g) of urea is sublimated in 250 mL of ethanol as a solvent for two hours. The reaction is monitored using (TLC). After the reaction is complete, the reaction was then cooled and cold water is added to the reaction mixture. The reaction mixture was neutralized by adding a 5% of sodium acetate. The resulting precipitate is filtered and recrystallized from ethanol [11].

4-(4-Nitrophenyl)oxazol-2-amine(1)

This compound resulted in 67% as a orange powder (m.p. 224-226 °C) , FT-IR (ν , cm^{-1}) 3483 (N-H), 3410, 2854 (C-H) alp., 1691 (C=N), 1522 (C-NO₂)_{Asym.}, 1342, (C-NO₂)_{Sym.}, 1171(C-O-C) _{Asym.}, 1006 (C-O-C)_{Sym.}. $^1\text{H NMR}$ (ppm): (DMSO d_6 , 400 MHz) δ : 7.83- 8.30 (m, 4H), 7.83(S, 1H), 5.41 (S, 2H). $^{13}\text{C NMR}$ (ppm): (DMSO d_6 , 400 MHz) δ : 125.23, 126.61, 127.97, 131.67, 132.31, 147.67, 145.3. CHNS elemental analysis calculated for C₉H₇N₃O₃: C, 52.66%; H, 3.44%; N, 20.48%. Found: C, 52.35%; H, 3.26%; N, 20.21%.

4-(4-Aminophenyl)oxazol-2-amine (2)

This compound resulted in 52% as a brown powder (m.p. 126-128 °C), FT-IR (ν , cm^{-1}) 3390, 3329 (N-H), 1650 (C=N), 1176 (C-O-C)_{Asym.}, 1023 (C-O-C)_{Sym.}. $^1\text{H NMR}$ (ppm): (DMSO d_6 , 400 MHz) δ : 6.57, 6.59, 7.64 (m, 4H), 7.67 (s, 1H), 5.32(s, 2H). $^{13}\text{C NMR}$ (ppm): (DMSO d_6 , 400 MHz) δ : 116.98, 127.29, 121.29, 131.0, 148.11, 157.13. CHNS elemental analysis calculated for C₉H₉N₃O: C, 61.70%; H, 5.18%; N, 23.99%; Found: C, 60.31%; H, 5.02%; N, 23.67%.

4-(Furan-2-yl) oxazol-2-amine (3)

This compound resulted in 75% as a brown powder (m.p 89-93°C), FT-IR (ν , cm^{-1})3549(N-H), 1689(C=N), 1169(C-O-C)_{Asym.}, 1021(C-O-C)_{Sym.}. $^1\text{HNMR}$ (ppm): (DMSO d_6 , 400 MHz) δ :6.56-7.39 (m, 4H), 7.40(s, 1H), 5.40(s, 2H). $^{13}\text{CNMR}$ (ppm): (DMSO d_6 , 400 MHz) δ : 112.19, 113.02, 114.24, 126.24, 130.51, 148.14, 157.42. CHNS elemental analysis calculated for C₇H₆N₂O₂: C, 56.00%; H, 4.03%; N, 18.66%; Found: C, 55.89%; H, 3.86%; N, 18.41%.

General procedure of preparation of complexes in a ratio of (2:1) (4-9)

In 100 mL round bottom flask dissolved 2 mmol of the prepared ligands in 25 mL of absolute ethanol and added to it a solution consisting of dissolving 1 mmol of the metal salt (CoCl₂.6H₂O)

or (PtCl₄) in 10 mL of absolute ethanol and added 1 mmol, 0.14 ml to the mixture of triethylamine dissolved in 10 mL of absolute ethanol. The mixture heated with stirring for two hours. The solution was cooled, and the resulting precipitate was filtered and recrystallized from ethanol [12].

Complex (4). It was prepared from ligand (1) with (CoCl₂.6H₂O) and result in 52% as a dark brown powder (m.p. 289^d °C), FT-IR (v, cm⁻¹) 3410 (N-H), 3362, 1691 (C=N), 1520 (C-NO₂)_{Asym.}, 1342 (C-NO₂)_{Sym.}, 1101 (C-O-C)_{Asym.}, 1006 (C-O-C)_{Sym.}, 502 (Co-N) and 410 (Co-O). CHNS elemental analysis calculated for C₁₉H₁₇Cl₂N₆O₆Co: C, 41.10%; H, 3.09%; N, 15.14%; Co, 10.61. Found: C, 40.90%; H, 2.90%; N, 15.14%; Co, 10.42%.

Complex (5). It was prepared from ligand (2) with (CoCl₂.6H₂O) and result in 61% as a dark brown powder (m.p. 289^d °C), FT-IR (v, cm⁻¹) 3359 (N-H), 3107, 1662 (C=N), 1101 (C-O-C)_{Asym.}, 1006 (C-O-C)_{Sym.}, 436 (Co-N) and 421 (Co-O). CHNS elemental analysis calculated for C₁₉H₂₁Cl₂N₆O₂Co: C, 64.08%; H, 4.27%; N, 16.97%; Co, 11.90. Found: C, 45.89%; H, 4.02%; N, 16.97%; Co, 11.78.

Complex (6). It was prepared from ligand (3) with (CoCl₂.6H₂O) and result in 53% as a yellow powder (m.p. 255^d °C), FT-IR (v, cm⁻¹) 3523 (N-H), 1671 (C=N), 1159 (C-O-C)_{Asym.}, 1009 (C-O-C)_{Sym.}, 431 (Co-N) and 402 (Co-O). CHNS elemental analysis calculated for C₁₅H₁₅Cl₂N₄O₄Co: C, 60.47%; H, 3.40%; N, 12.59%; Co, 13.24%. Found: C, 40.21%; H, 3.31%; N, 12.37%; Co, 13.24%.

Complex (7). It was prepared from ligand (1) with (PtCl₄) and result in 42% as an Brown powder (m.p. 260^d °C), FT-IR (v, cm⁻¹) 3469 (N-H), 3394, 1681 (C=N), 1538 (C-NO₂)_{Asym.}, 1361 (C-NO₂)_{Sym.}, 1151 (C-O-C)_{Asym.}, 1012 (C-O-C)_{Sym.}, 449 (Pt-N) and 421 (Pt-O). CHNS elemental analysis calculated for C₁₈H₁₄Cl₄N₆O₆Pt: C, 28.93%; H, 1.89%; N, 11.25%; Pt, 26.11 %. Found: C, 28.71%; H, 1.65 %; N, 11.0 2 %; Pt, 28.51.

Complex (8). It was prepared from ligand (2) with (PtCl₄) and result in 41% as an brown powder (m.p. 289^d °C), FT-IR (v, cm⁻¹) 3385 (N-H), 3330, 1650 (C=N), 1174 (C-O-C)_{Asym.}, 1071 (C-O-C)_{Sym.}, 456 (Pt-N) and 412 (Pt-O). CHNS elemental analysis calculated for C₁₈H₁₈Cl₄N₆O₂Pt: C, 31.46 %; H, 2.64 %; N, 12.23 %; Pt, 28.39%. Found: C, 31.13 %; H, 2.81 %; N, 12.12 %; Pt, 31.48%.

Complex (9). It was prepared from ligand (3) with (PtCl₄) and result in 44% as an brown powder (m.p. 280^d °C), FT-IR (v, cm⁻¹) 3362 (N-H), 3105, 1689 (C=N), 1109 (C-O-C)_{Asym.}, 1060 (C-O-C)_{Sym.}, 486 (Pt-N) and 430 (Pt-O). CHNS elemental analysis calculated for C₁₄H₁₂Cl₄N₄O₄Pt: C, 26.39%; H, 1.90%; N, 8.79%; Pt, 30.62%. Found: C, 26.22 %; H, 1.79 %; N, 8.51 %; Pt, 30.50%.

Biological study

A study was conducted to examine the impact of many produced compounds on four different types of bacteria, involving both Gram-negative and Gram-positive bacteria. The bacteria species considered were *Escherichia coli*, *Staphylococcus aureus*, *Klebsiella pneumonia*, and *Salmonella typhi*. The positive and negative bacteria were gained from the laboratory of the Life Sciences Department/College of Education for Pure Sciences, University of Mosul.

Inhibitory efficacy test

The Levne method [13], which is copied from the Vandepitte method [14], was retained. This include injecting the Nutrient Saline medium with individual colonies of the bacteria indicated

before, followed by incubation. The bacteria were cultured at a temperature of 37 °C for a period of 18-24 hours. Following this, a series of dilutions were accomplished using normal saline solution to achieve a concentration of 10⁸ cells/cm³, which was later compared to the concentration in tube No. 1 (Macfarland standard tubes 1) The bacterial suspension was applied onto the standard nutritional agar surface using a sterile glass diffuser. The plates were then incubated for 30 min to let imbibition to take place.

To find the antibacterial activity of the synthesised compounds, circular filter paper discs measuring 6 mm in diameter were made. These discs were then soaked with specified quantities of the compounds dissolved in dimethyl sulfoxide, which were precisely chosen. The discs were subsequently affixed onto the surface of the agar plates using sterile forceps and placed in an incubator at a temperature of 37 °C for a duration of 18-24 hours. Following incubation, the diameter of inhibition was measured, and certain plates were compared to standard antibiotics (Ampicillin) as control samples.

RESULTS AND DISCUSSION

Preparation of oxazoles compounds

2-Amino oxazole (**1-3**) compounds were prepared by the reaction of 4-nitroacetophenone, 4-aminoacetophenone, or 2-acetylfuran respectively with urea in ethanol as a solvent (Scheme 1). The infrared spectrum showed frequency bands in the range (1691-1650 cm⁻¹) that belong to the frequency of the stretch group (C=N) in the oxazole ring. These frequencies did not have a clear change after coordination with the metal ion, and this confirms that they do not participate in coordination, as the spectrum gave absorption frequency bands in the range (3593-3393 cm⁻¹) that belong to the stretch bands of the (N-H) group. After coordination with the metal ion, it was observed that the frequency was shifted to a lower wavelength, indicating the participation of the electron pair on the nitrogen atom in the formation of the complex, and the appearance of bands. At a frequency of (375 cm⁻¹), it was indicated that the metal is compatible with oxygen (M-O) [15, 16].

The ¹H-NMR spectrum of the prepared compounds was also studied for (**1**) gave a multiple signal of medium intensity at the site (5.41 ppm) belonging to the (2H) protons of the NH₂ group, and another single signal of at (7.83 ppm) belongs to the (1H) protons of the carbon atom of the oxazole ring adjacent to the oxygen atom. In addition, two doublet signals belong to the (4H) of the symmetric aromatic ring at (7.83-8.30 ppm).

The ¹³C-NMR chart of compound (**1**) showed signals at the positions (125.23, 127.97 ppm) belonging to the four symmetric carbon atoms of the aromatic ring. It also showed a band at (131.67 ppm) belonging to the carbon atom of the oxazole ring is adjacent to the oxygen, and a band at the position (132.31 ppm) belongs to the carbon atom of the oxazole ring attached to the phenyl, in addition, a band at the position (147.67 ppm) goes back to the carbon atom of the phenyl ring attached to the NO₂ group, and another band at the position (154.31 ppm) belong to the carbon atom of the oxazole ring attached to the NH₂ group. (Figure 1).

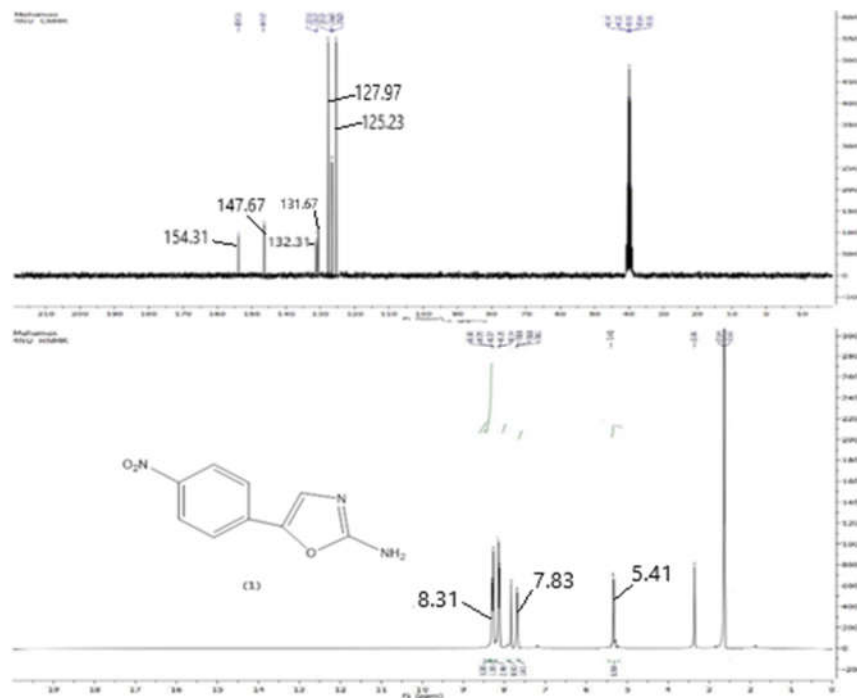


Figure 1. ^1H NMR and ^{13}C NMR spectra of **1**.

The proton nuclear magnetic resonance spectrum. (^1H -NMR) of compound (**2**) showed a distinct, singlet signal at the site (5.32 ppm) belong to the (2H) proton of the terminal NH_2 group attached to the oxazole ring, as well as a single signal at (4.10 ppm) belong to two protons of the NH_2 group attached to the phenyl ring. In addition, two doublet signals at (7.67, 6.59 ppm) belong to four symmetrical protons of the aromatic ring, and another single signal at t (7.23 ppm) goes back to the one proton of the carbon atom of the oxazole ring adjacent to the oxygen atom.

The ^{13}C -NMR spectrum of compound (**2**) gave two signals at (116.18, 131.05 ppm) belonging to the four symmetrical carbons in phenyl rings and a signal at (148.11 ppm) goes back to the carbon atom linked to the NH_2 group. Another signal at the (121.29 ppm) belong to the carbon linked to the oxazole group. The oxazole ring gave signals at (127.29 ppm) goes back to the carbon atom next to the oxygen. Another signal at the position (158.58 ppm) that goes back to carbon atom attached to the NH_2 group (Figure 2).

The formula of compound (**3**) was also confirmed by the nuclear magnetic resonance (^1H -NMR) spectrum, as it gave singlet signal at (5.40 ppm) belong to two proton of the NH_2 group, Also there are signals (6.56-7.39 ppm).) belong to three protons of the furan ring. In addition, there is a singlet signal at (7.40 ppm) back to one proton of the carbon atom adjacent to the oxygen atom in the oxazole ring [17, 18].

The ^{13}C -NMR spectrum of compound (**3**) showed signals at (112.19, 113.02, 144.24 ppm) belonging to the three unsubstituted carbon atoms of furan ring. It also showed signal at the positions (126.24 ppm) belonging to the carbon atoms linked to the oxazole ring. it also showed a band at the position (130.51 ppm) go back to the carbon atom of the oxazole ring next to the oxygen, and a band at the position (148.14 ppm) belong to the carbon atom of oxazole ring linked

to furan. In addition, a band at position (157.42) ppm refers to the carbon atom of the oxazole ring attached to the NH₂ group [19] (Figure 2) (Scheme 1).

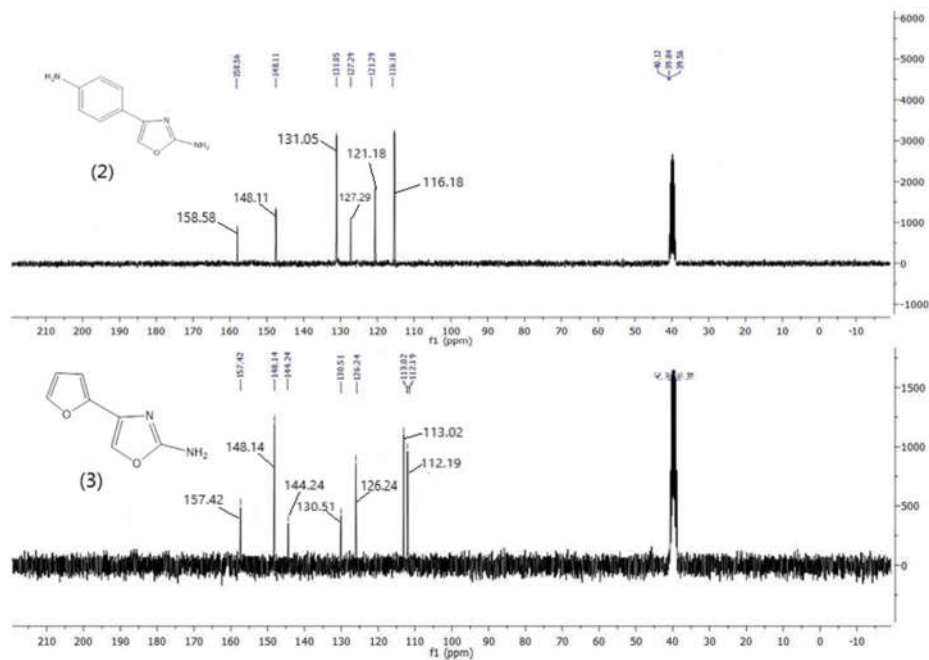
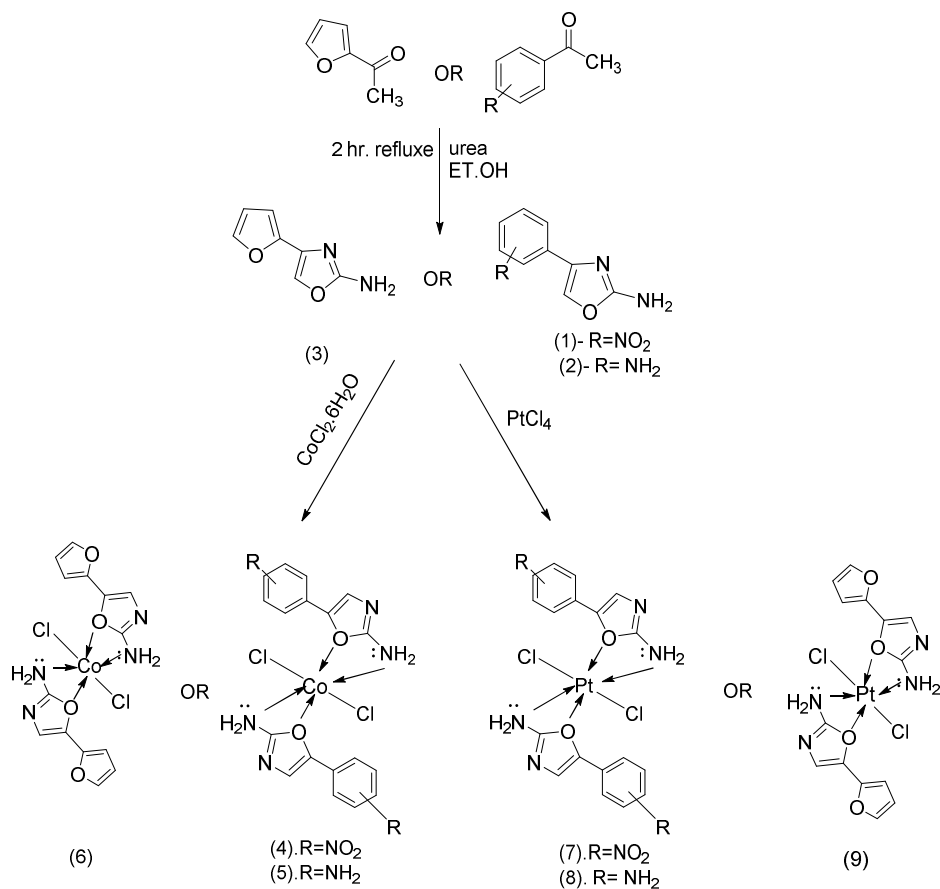


Figure 2. The ¹³C NMR spectra of (2, 3).



Scheme 1. Total synthesis reactions.

Magnetic measurements and electronic spectra

The prepared cobalt(II) complexes that possess the cobalt(II) system (d^7) gave the electronic arrangement $t^2g^5 eg^2$ and have magnetic moment values ranging (3.96-4.83) B.M., which is due to the presence of three individual electrons in the (d^7) system. The value of the effective magnetic moment is due to the value higher than the theoretically calculated value indicates the presence of the orbital contribution, and this is consistent with the values of the magnetic moment for the hexagonal cobalt(II) complexes with a highly twisted octahedral geometric shape [20, 21].

As for the prepared platinum(IV) complexes, which possess the platinum(IV) (d^6) system, which has the electronic arrangement $t^2g^5 eg^1$, they have diamagnetic properties, with a magnetic moment ranging between (1.02-1.1) and have a hexagonal octahedral structure [22].

The electronic spectra of the cobalt(II) complexes shown in Table 1 that the complexes (4-6) have three bands, the first band ν_1 is located in the range (12845-15728) cm^{-1} and the second band ν_2 is in the range (17421-18150) cm^{-1} and the third band ν_3 (24941-30472) cm^{-1} of the spectrum, in addition to the charge transfer band, which appears above cm^{-1} (30000). The

appearance of bands ν_1 , ν_2 and ν_3 confirms that these transitions are permissible. It goes back to octahedral cobalt complexes (Table 1) [23].

Table 2 shows the electronic spectra of platinum (IV) complexes (7-9) have three bands, the first band is located ν_1 (13856-13978) cm^{-1} and the second band is ν_2 (16116-24156) cm^{-1} and the third is ν_3 (23256-24156) cm^{-1} in addition to the charge transfer band, which appears above (30000) cm^{-1} . The appearance of these four transitions indicates the symmetry of the platinum(IV) complexes octahedral [24].

Table 1. The electronic spectra and magnetic properties of cobalt(II) complexes.

Compound No.	Legand No.	${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{2g}(\text{F})$ ν_1	${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{A}_{2g}(\text{F})$ ν_2	${}^4\text{T}_{1g}(\text{F}) \rightarrow {}^4\text{T}_{1g}(\text{P})$ ν_3	C.T (cm^{-1})	μ_{eff} B.M.	Suggested structure
4	1	14673	18008	30120	34655	4.83	Oh
5	2	15728	17421	30472	35137	4.62	Oh
6	3	12845	18150	24941	34081	3.96	Oh

Table 2. The electronic magnetic properties spectra of platinum(IV) complexes.

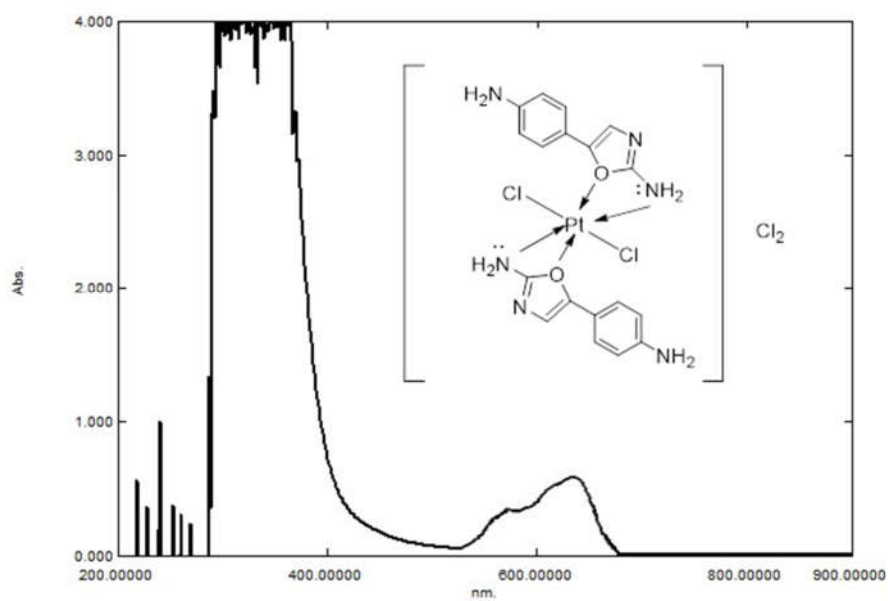
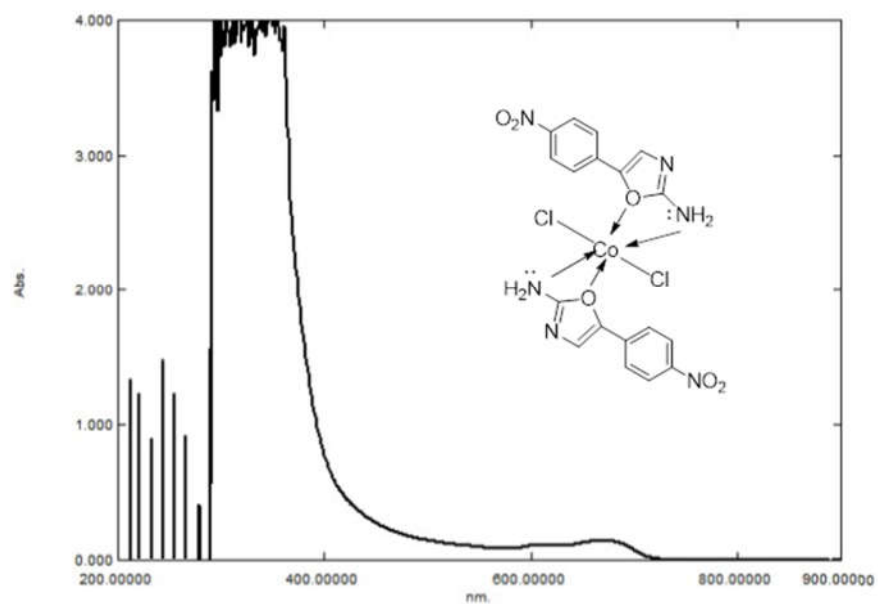
Compound No.	Legand No.	${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{1g}$ ν_1	${}^1\text{A}_{1g} \rightarrow {}^3\text{T}_{2g}$ ν_2	${}^1\text{A}_{1g} \rightarrow {}^1\text{T}_{1g}$ ν_3	C.T. (cm^{-1})	μ_{eff} B.M.	Suggested structure
7	1	13856	16231	23256	36213	Dia.	Oh
8	2	13891	16123	24156	36276	Dia.	Oh
9	3	13978	16116	23496	36114	Dia.	Oh

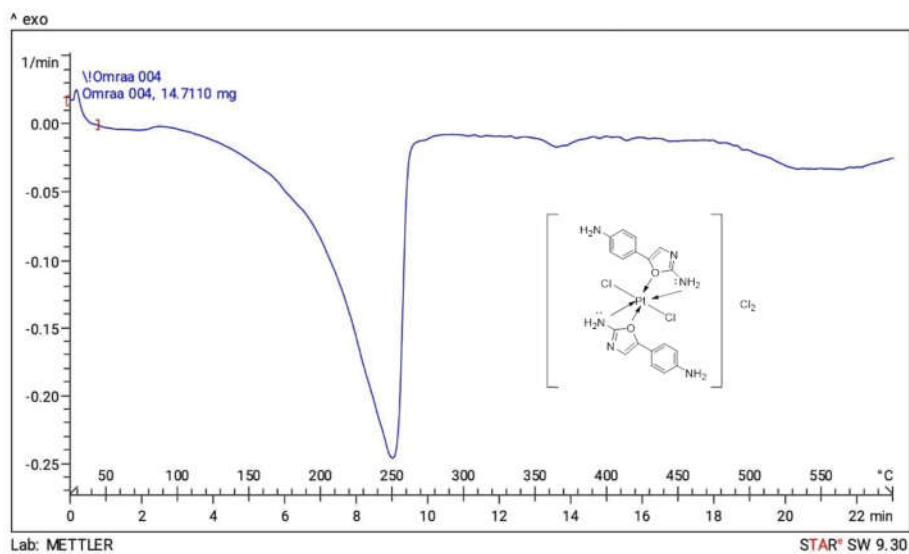
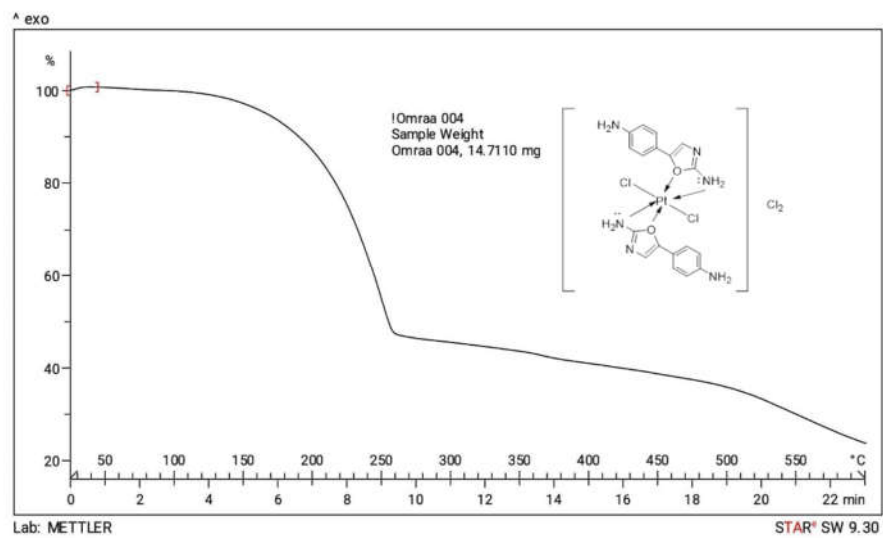
The molar electrical conductivity of the prepared complexes was measured at a concentration (10^{-3} M) using the solvent dimethyl sulfoxide (DMSO). It was found from the molar electrical conductivity measurements that it agrees with the proposed structural formulas for the prepared complexes of the type (1:2), as it was found that cobalt complexes. All of the preparations fall within the range of complexes with neutral behavior, non-electrolytes or weak conductors, and the molar electrical conductivity values of these complexes in the solvent (DMSO) ranged between (1.5-16.0) ($\text{cm}^2 \cdot \text{ohm}^{-1} \cdot \text{mol}^{-1}$). [25].

The prepared platinum complexes of the type (1:2) fall within the category of complexes with electrical conductive behavior, and the molar electrical conductivity values of these complexes in the solvent (DMSO) ranged between (70.1-79.2) ($\text{cm}^2 \cdot \text{ohm}^{-1} \cdot \text{mol}^{-1}$). The results show existence of two chlorine atoms outside of the coordination sphere [26].

Differential and gravimetric thermal analysis of complexes

Thermogravimetric and differential analysis of the compound (8) was studied, and we notice through the TGA that the complex began to lose weight after a temperature of 150 °C, and this indicates that the complex does not contain water molecules, in addition, at a temperature of 250 °C, the loss rate reached approximately 50% resulting regarding the process of changing from solid to liquid or the beginning of the disintegration of the ligand, the percentage of weight loss remained approximately this percentage up to a temperature of 500 °C, and at a temperature of 550, Therefore, the remainder of the thermal and gravimetric decomposition process is atoms in the form of metal oxides with a small amount of black carbon [27] the percentage of loss reached 80%. What is worth noting is the correspondence and compatibility of these changes with differential thermal analysis (DTA) [28] (Figure 3).





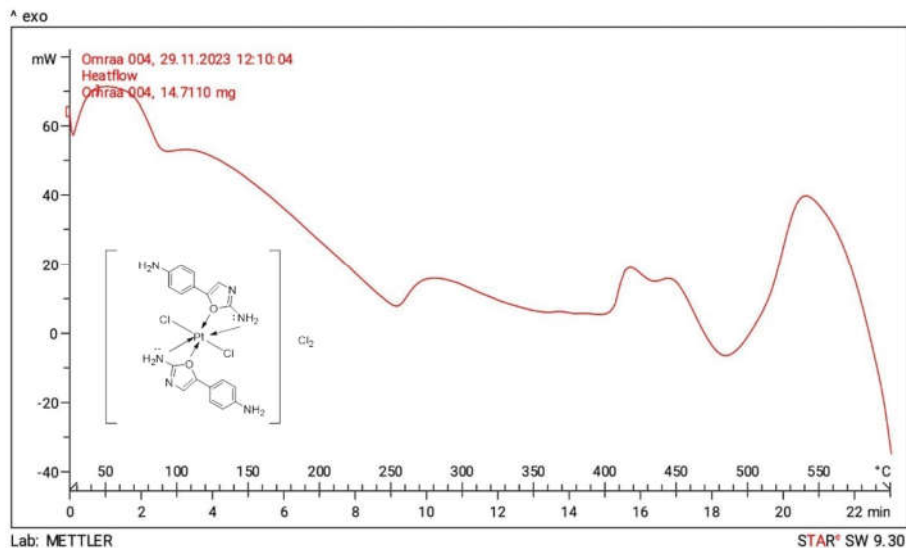


Figure 3. TGA and DTA curves and UV-Visible curves.

Biological effectiveness

The biological effect of a number of prepared compounds (**2**, **5**, **6**, **7**, **8**, **9**) was tested against four types of Gram- negative and Gram- positive bacteria, namely *Staphylococcus aureus*, *Escherhsia coli*, *Klebsiella pneumonia*, and *Salmonella typhi*.

Using the disc diffusion method, these bacteria were chosen due to their importance in the medical field and the fact that they cause a number of diseases and differ in the nature of their resistance to antibiotics and various medications. The inhibitory results shown in Table 3 indicate that some of the prepared compounds have potent activity to inhibit the bacteria. According of the inhibitor zone the complexes (**8**, **9**) gave excellent inhibitory against *E. coli* and *Pneumonia klebsiella* than the ampicillin as a control [29]. While other compound fluctuated between moderate to good and weak just in one case for (**2**) against *Salmonella typhi* and *Klebsiella pneumonia*.

Table 3. Biological activity.

Compound No.	<i>Staphylococcus aureus</i> 10 (mg/mL) *ZI mm	<i>Escherhsia coli</i> 10 (mg/mL) ZI mm	<i>Klebsiella pneumonia</i> 10 (mg/mL) ZI mm	<i>Salmonella typhi</i> 10 (mg/mL) ZI mm
2	13	11	6	0
5	13	11	14	8
6	20	12	10	7
7	24	10	11	8
8	12	24	26	22
9	20	21	24	25
Ampicillin 10 mg/disk	40	20	10	20

*ZI = zone inhibitor in millimetre.

CONCLUSION

In this study, we synthesized three different substituted 2-aminoxazoles by reacting substituted acetophenone or acetyl furan with urea. These synthesized compounds served as ligands to form six complexes with metals, specifically Co(II) and Pt(IV). We performed thermal analysis (TGA), magnetic property assessments, and differential thermal analysis (DTA) on these complexes. Additionally, the biological activity of the synthesized compounds was evaluated, showing good to excellent effectiveness against the selected bacteria.

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