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RESEARCH ARTICLE

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The ligational behavior of an isatinic quinolyl hydrazone towards copper(II)- ions

Hussein S Seleem*, Gaber A El-Inany, Bashir A El-Shetary, Marwa A Mousa and Fatin I Hanafy

Abstract

Background: The importance of the isatinic quinolyl hydrazones arises from incorporating the quinoline ring with the indole ring. Quinoline ring has therapeutic and biological activities whereas, the indole ring occurs in Jasmine flowers and Orange blossoms. As a ligand, the isatin moiety is potentially ambidentate and can coordinate the metal ions either through its lactam or lactim forms. In a previous study, the ligational behavior of a phenolic quinolyl hydrazone towards copper(II)- ions has been studied. As continuation of our interest, the present study is planned to check the ligational behavior of an isatinic quinolyl hydrazone.

Results: New homo- and heteroleptic copper(II)- complexes were obtained from the reaction of an isatinic quinolyl hydrazone (HL) with several copper(II)- salts *viz.* Cl^-, Br^-, NO_3^-, ClO_4^-, SO_4^{2-} and AcO^-. The obtained complexes have O_h, T_d and D_{4h}- symmetry and fulfill the strong coordinating ability of Cl^-, Br^-, NO_3^- and SO_4^{2-} anions. Depending on the type of the anion, the ligand coordinates the copper(II)- ions either through its lactam (NO_3^- and ClO_4^-) or lactim (the others) forms.

Conclusion: The effect of anion for the same metal ion is obvious from either the geometry of the isolated complexes (O_h, T_d and D_{4h}) or the various modes of bonding. Also, the obtained complexes fulfill the strong coordinating ability of Cl^-, Br^-, NO_3^- and SO_4^{2-} anions in consistency with the donor ability of the anions. In case of copper(II)- acetate, a unique homoleptic complex (**5**) was obtained in which the AcO^- anion acts as a base enough to quantitatively deprotonate the hydrazone. The isatinic hydrazone uses its lactim form in most complexes.

Background

Hydrazones and their metal complexes present innumerous pharmacological applications as antimicrobial, anticonvulsant, analgesic, anti-inflammatory, anti-tubercular and antitumor agents [1-3]. Also, the metal complexes of hydrazones have potential applications as catalysts [4], luminescent probes [5] and molecular sensors [6]. The importance of the isatinic quinolyl hydrazones arises from incorporating the quinoline ring with the indole ring. Quinoline ring has therapeutic and biological activities whereas, the indole ring occurs in Jasmine flowers and Orange blossoms[1-3]. As a ligand, the isatin moiety is potentially ambidentate and can coordinate the metal ions either through the lactam or lactim forms. In general, this study is planned to investigate the ligational behavior of the studied hydrazone (Scheme 1); 3-[2-(4,8-dimethylquinolin-2-yl)hydrazono]indolin-2-one (HL) towards several copper(II)- salts (Cl^-, Br^-, NO_3^-, ClO_4^-, SO_4^{2-} and AcO^-). This work is an extension to our previous studies on the chelating ability of some quinolyl hydrazones [7-11].Scheme 1 Tautomeric forms of the isatinic hydrazone.

Results and discussion

Characterization of the hydrazone

The investigated hydrazone (HL) was prepared by a condensation reaction of 2-hydrazinyl-4,8-dimethylquinoline with isatin (indol-2,3-dione). The results of elemental analysis (Table 1) are in good harmony with the proposed formula. The IR spectrum (Table 2) showed very strong bands at 3203 and 1605 cm^{-1} which are assigned to ν(NH) and ν(C=N), respectively. The lactam nature of HL was supported by a very strong band at 1706 cm^{-1}; ν(C=O). On the other hand, the electronic absorption spectra of the hydrazone in DMF exhibit two intense bands at 268 and 391 nm characteristic for π-π* and charge transfer (CT) transitions. The latter band

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* Correspondence: hsseleem@yahoo.com
Department of Chemistry, Faculty of Education, Ain Shams University, Roxy, Cairo, Egypt

Table 1 Analytical and physical data of the copper(II)- isatinic complexes.

No.	Reactants (HL + metal salt)	Complex (F.W.)	Color	% Yield	m.p. (°C)	Elemental Analysis; % Found/(Calcd.)			
						C	H	N	M
	HL	HL ($C_{19}H_{16}N_4O$; 316.36)	Red	60	> 300	72.06 (72.13)	5.17 (5.10)	17.70 (17.71)	——
1	Cu $(ClO_4)_2.6H_2O$	[Cu (L) (HL) $(H_2O)_2$] $ClO_4.3H_2O$ (884.79)	Red	60	> 300	51.54 (51.58)	4.63 (4.67)	12.69 (12.67)	7.25 (7.18)
2	Cu $(NO_3)_2.21/2H_2O$	[Cu $(HL)_2$ $(NO_3)_2$].1¼ H_2O (842.81)	Yellowish brown	61	> 300	54.10 (54.15)	4.12 (4.13)	16.44 (16.62)	7.62 (7.54)
3	Cu $Cl_2.2H_2O$	[Cu (L) (H_2O) Cl].1/2 H_2O.⊠ MeOH (445.38)	Dark green	30	252	51.48 (51.57)	4.14 (4.19)	12.57 (12.58)	14.10 (14.27)
4	Cu Br_2	[Cu (L) $(H_2O)_3$ Br].⊠ H_2O (519.60)	Granulated greenish brown	60	> 300	43.92 (43.92)	4.15 (4.22)	10.81 (10.79)	12.40 (12.23)
5	Cu $(OAc)_2.H_2O$	[Cu $(L)_2$].⊠ H_2O.⊠ MeOH (700.51)	Bright greenish brown	82	292	65.47 (65.36)	4.51 (4.42)	15.93 (15.99)	8.90 (9.07)
6	Cu $SO_4.5H_2O$	[Cu_2 $(L)_2$ $(H_2O)_4$ SO_4].2 H_2O.MeOH (994)	Dark orange	64	> 300	47.12 (47.12)	4.69 (4.66)	11.13 (11.28)	12.70 (12.79)

(CT) impact the ligand its red color whereas, the higher energy band is consistent with that reported for the aromatic quinoline ring [7-11]. The mass spectrum of the ligand showed the M^+ peak at *m/z* = 316 confirming its formula weight (316.36). The mass fragmentation pattern (Scheme 2) supported the suggested structure of the ligand. Finally, the ^1H NMR spectral data of the ligand in d_6-DMSO relative to TMS; Figure 1, lend a further support of the structure.Scheme 2

Mass fragmentation pattern of the isatinic hydrazone.

Characterization of the isatinic complexes

The isatinic hydrazone (HL) has mainly two tautomeric forms (Scheme 1). Both rearrangements; the lactam-lactim forms are useful in explaining the different coordinating properties. The ligand was allowed to react with several copper(II)- salts *viz.* Cl^-, Br^-, NO_3^-, SO_4^{2-}, ClO_4^- and AcO^- in order to determine the effect of the anions on the formed products (Scheme 3). The obtained copper(II)- complexes reflect the strong coordinating power of SO_4^{2-}, Cl^-, Br^- and NO_3^- as compared to the weakly or non coordinating power of ClO_4^-. This is consistent with the donor ability of the anions [12-14]; (DN_x = 36.2, 33.7, 21.1 and 8.44 for Cl^-, Br^-, NO_3^- and ClO_4^-, respectively). All complexes are mononuclear except complex **6** which is binuclear. The obtained complexes have the octahedral (O_h), tetrahedral (T_d) and square planar (D_{4h}) geometry; (Scheme 3) and reflect the non participation of the heterocyclic N- atom of the quinoline ring in the chelation. The isatinic hydrazone (HL) behaves as monoanionic or neutral NO- donor which is consistent with steric properties of the more rigid ligand. Characterization of the obtained complexes was achieved *via* elemental and thermal analyses, magnetic and conductivity measurements as well as spectral studies.Scheme 3

Effect of anion on the copper(II) - isatinic complexes.

IR spectra of the complexes

The mode of bonding was studied by comparing the IR spectral bands of the metal complexes with those of the free ligand (Table 2). Inspection of the data revealed the following: (i) All complexes showed a broad band in the range 3463-3203 cm^{-1} due to ν(OH) of the associated water or methanol molecules. (ii) The band at 1605 cm^{-1} assignable to ν(C=N) in the free ligand was shifted to higher values indicating the participation of C=N of the hydrazone moiety in the chelation with π- electron

Table 2 Magnetic, conductivity, electronic and IR spectral data of the copper(II)- isatinic complexes.

Complex	λ (nm)	μ_{eff} B.M.	Conductance Ω^{-1} cm^2 mol^{-1}	IR spectral bands; cm^{-1}			
				ν (NH)	ν (C = O)	ν (C = N)	Others
HL	268, 391	———	——	3203	1706	1605	————
1	396	1.80	95.0	3287	1653	1627	1100; ν_3(ClO)
2	396	1.93	12.0	3463	1692	1623	1387 and 1297; ν (NO)
3	391	1.87	37.0	3235 br.	———	1619	————
4	392	1.82	40.0	3203 br.	———	1621	————
5	453	1.77	Zero	3363	———	1608	————
6	450	1.40	15.0	3273	———	1633	1135; ν_3 (SO)

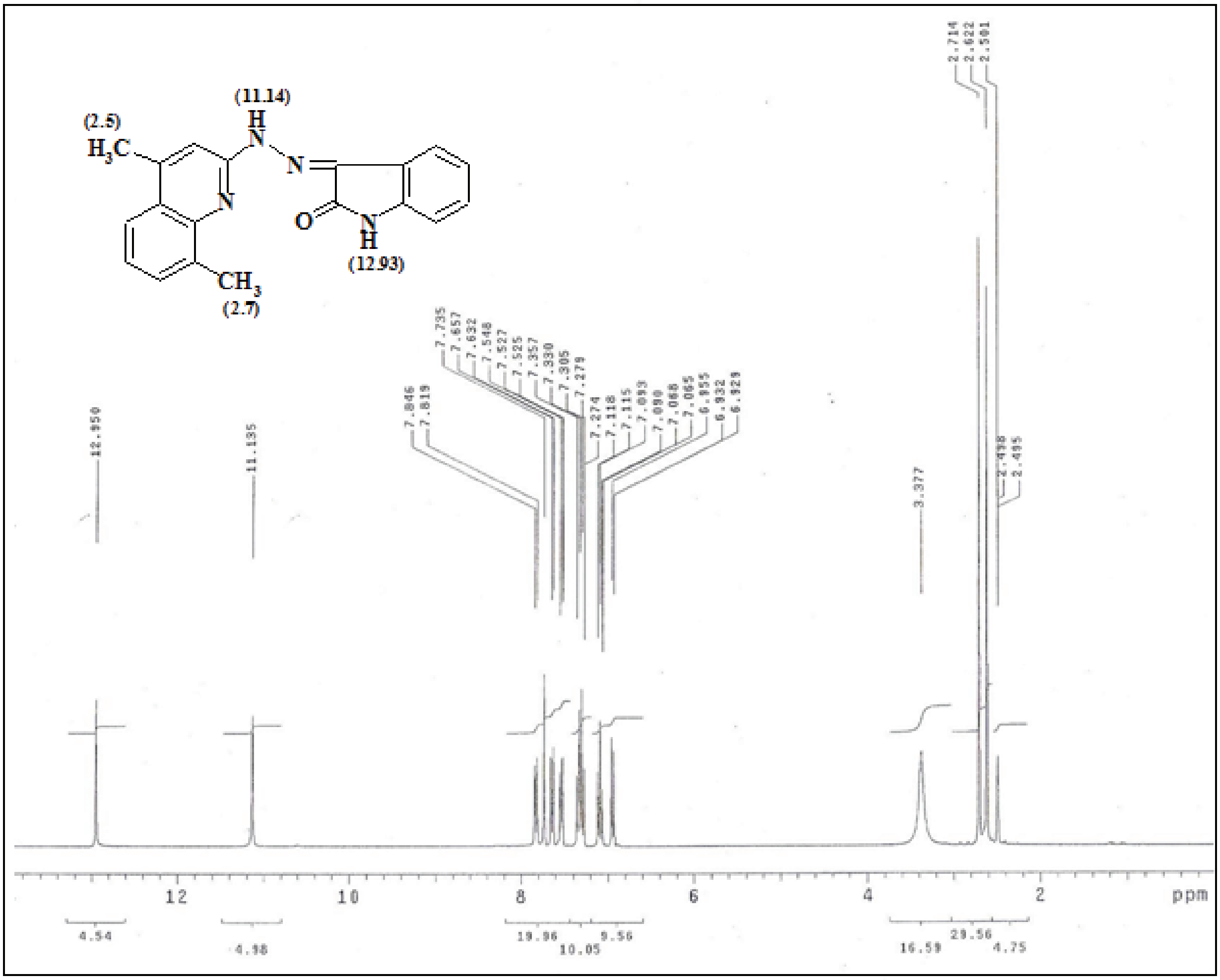

Figure 1 ^{1}HNMR spectrum of the isatinic hydrazone; δ = 6.96-7.74 for aromatic protons (8H).

delocalization. (iii) In most complexes, the band located at 1706 cm^{-1} due to ν(C=O) of the free ligand disappeared indicating the participation of the lactim- form in the chelation. In contrast, the lactam- form participates in the chelation in case of the nitrato (**2**) and perchlorato (**1**) complexes as indicated by the shift of the above band to lower wave numbers; 1692 and 1653 cm^{-1}, respectively. However, the greater lower value 1653 cm^{-1} for the perchlorate complex (**1**) may suggest mixed modes of bonding (lactim + lactam) [7]; Scheme 3. (iv) In complex **1**, the strong broad band centered at 1100 cm^{-1} (antisymmetric stretch) and the sharp band at 621 cm^{-1} (antisymmetric bend) suggest uncoordinated ClO_4^- anion [14]. (v) For the binuclear sulfato complex (**6**), the chelating bidentate nature of the SO_4^{2-} group is indicated by the appearance of ν_3(S-O) strong band at 1135 cm^{-1} characteristic for the high symmetry T_d (tetrahedral) point group. The nitrato complex (**2**) showed two bands at 1387 and 1297 cm^{-1} confirming the monodentate nature of the coordinated NO_3^- group; C_{2v} symmetry. (vi) Finally, the detection of the non ligand bands; ν(M-O) and ν(M-N) in the finger print region is more difficult and tentative.

Conductivity and magnetic properties

The recorded conductance for 10^{-3} molar DMF solutions of the complexes (Table 2) indicates that all complexes are non-conducting due to their neutrality (Λ = 17.0-1.9 Ω^{-1} cm^2 mol^{-1}). The only exception is $[Cu(L)(HL)(H_2O)_2]ClO_4.3H_2O$ (**1**) which showed molar conductance of 95 Ω^{-1} cm^2 mol^{-1}, indicating its 1:1 electrolytic nature which is consistent with the IR spectra; ν_3 (Cl-O) at 1100 cm^{-1}. In contrast, the halo-complexes (**3** and **4**) showed molar conductance of 37 and 40 Ω^{-1} cm^2 mol^{-1}, respectively indicating their partial electrolytic nature which is due to the replacement of the coordinated Cl$^-$ and Br$^-$ ions by DMF solvent molecules [4]. On the other side, the effective magnetic moments (μ_{eff})

of the copper(II)- complexes (**1**-**5**) lie in the range 1.93-1.77 B.M. (Table 2) which is consistent with one unpaired electron and falls within the range reported for mononuclear copper(II)- complexes. However, the binuclear copper(II)- complex (**6**) exhibits lower μ_{eff} value at 1.4 B.M. indicating some metal—metal interaction.

Mass and electronic spectra

The mass spectra of complexes **3**; [Cu (L) (H_2O) Cl].1/2 H_2O.⅛ MeOH (445.38) and **4**; [Cu (L) $(H_2O)_3$ Br].⅜ H_2O (519.60) as representative examples showed their molecular ion and base peaks at m/e (447 and 156) and (519 and 45), respectively, confirming their suggested structures. On the other side, the electronic spectra of the complexes as DMF solutions showed a new intense broad band in the range 391-453 nm confirming its charge transfer (CT) nature (Figure 2 and Table 2). Therefore, the type of the d-d transitions can not be identified due to the strong CT bands tailing from UV region to the visible region. In general, the color of all complexes is dominated by the CT transition which obscured the weak d-d transition occurring in the same region; a phenomenon encountered with isatinic complexes [4]. Also, the electronic spectra of all complexes are nearly similar in terms of the position, intensity and shape of the bands (Figure 2 and Table 2).

Electron spin resonance spectroscopy

The ESR spectra of $[Cu(L)(HL)(H_2O)_2]ClO_4.3H_2O$ (**1**) and $[Cu (L)_2]$.⅛ H_2O.⅛ MeOH (**5**); Figure 3 are largely different, however, the ESR spectral profile of complex **5** confirms its T_d geometry. Generally, ESR spectra are useful for distinguishing square planar and tetrahedral geometries. The ESR spectral parameters were used to evaluate the bonding parameters α^2 and β^2 which may be regarded as a measure of the in-plane σ- bonding and the in-plane π- bonding, respectively. The following simplified expressions were used to evaluate the bonding parameters [15,16]:

$$\alpha^2 = [\mathrm{E}(g_{11} - g_e)]/8\nu\beta^2 \text{ and } \beta^2 = [E(g_\perp - g_e)]/2\nu\gamma^2$$

where E is the transition energy, ν represents the electron spin-orbit coupling and γ^2 represents the out-of-plane π-bonding and was taken as a constant value. On the other hand, the super-exchange splitting constant A_{11} was evaluated from the following expression:

$$\alpha^2 = (\frac{A_{11}}{P}) + (g_{11} - g_\perp) + \frac{3}{7}(g_\perp - g_e) + 0.04$$

where P is the free ion dipolar term and its value depends on the type of the metal ion. Also, the *f*- factor ($f = g_{11}/A_{11}$) was evaluated to determine the extent of tetragonal distortion. The obtained ESR parameters are shown in Table 3.

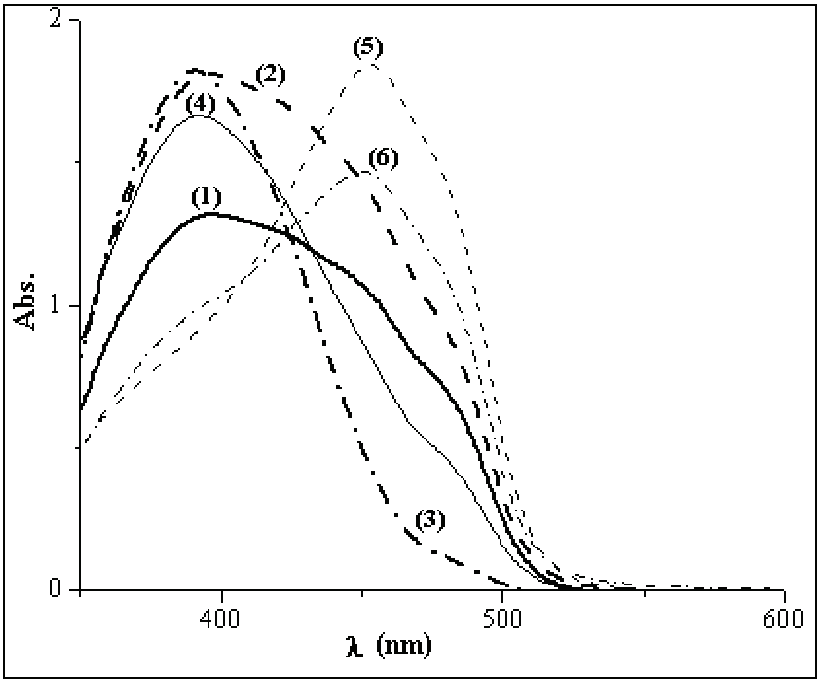

Figure 2 Overlay of the electronic spectra of the copper(II)-complexes.

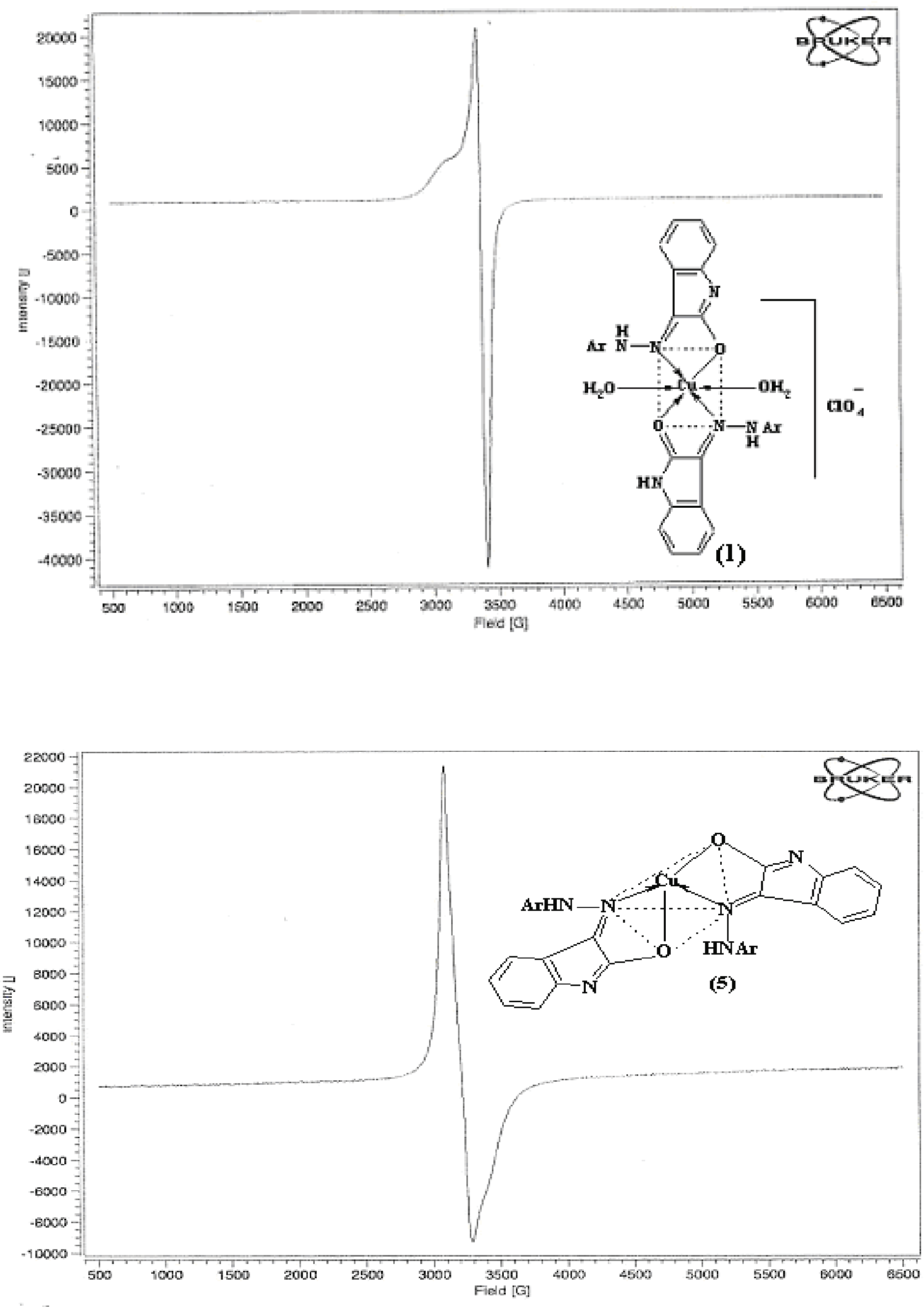

Figure 3 The X-band ESR spectra of complexes 1 (O_h) and 5 (T_d).

Table 3 ESR parameters

Complex	g_{11}	$g_{\perp}$	<g>	G	A^2	β^2	K_{Π}^2	$(A_{11})\text{cm}^{-1}$	*f*
(**1**); O_h	2.174	2.076	2.109	2.033	0.582	0.534	0.311	154×10^{-4}	141
(**5**); T_d	2.187	2.052	2.097	3.716	0.93	0.36	0.335	61×10^{-4}	362

In summary, the T_d- complex (**5**) with the lactim mode showed either higher values of G, α^2, K_{Π}^2 and f or lower values of β^2 and A_{Π} as compared to the O_h complex (**1**) with the mixed mode (lactam + lactim) which is consistent with the greater discrepancy of the observed spectra.

Thermal (TG-DSC) analysis

The thermal degradation behavior of the investigated complexes was followed by the thermogravimetric (TG) and differential scanning calorimetric (DSC) techniques. The decomposition occurs in one or more steps according to the nature of each complex. Attempts to generalize the thermal degradation patterns were unsuccessful indicating that there is no simple relation or general trend for explaining these thermal degradations. However, the decomposition ends with the formation of Cu_2O in most cases. Inspection of the TG thermograms revealed the following: (i) The perchlorato complex (**1**) decomposes in one strong endothermic step (ΔH = 391 J/g at 272°C) to form Cu_2O as the end product, a phenomenon encountered with ClO_4^- anions [14]. (ii) For the nitrato- (**2**) and sulfato (**6**)- complexes, the decomposition is not completed up to 800°C, indicating that the metal-ligand bonds are strong. This is consistent with the data extracted from IR. (iii) In contrast, for the other complexes(**1**, **4** &**5**), the decomposition process is ended with Cu_2O.

Kinetic and thermodynamic parameters

The activation energy (E*) of decomposition of the complexes as well as the pre-exponential factor (A) were evaluated graphically using the Coats-Redfern equation;

$$\ln\left[\frac{-\ln(1-\alpha)}{T^2}\right] = \frac{-E^*}{RT} + \ln\left[\frac{AR}{\phi E^*}\right]$$

where α is the fraction of sample decomposed at temperature T, A the pre-exponential factor, ϕ the heating rate, E* the activation energy and R the gas constant. A plot of ln [-ln (1 - α)/T^2] against 1/T gives a straight line whose slope = -E*/R and its intercept = ln [AR/ϕ E*]; a sample set (Figure 4). The activation entropy, enthalpy and free energy changes of the decomposition were calculated by the following equations.:

$$\Delta S* = 2.303\ R \log(Ah/KT),\ \Delta H* = E * -RT \text{ and } \Delta G* = \Delta H * -T\Delta S*$$

where h, K and T are the Planck's constant, Boltzmann's constant and the mid temperature in the TG-thermogram, respectively. The calculated values of A, E*, ΔS*, ΔH* and ΔG* for the decomposition steps are given in Table 4. Inspection of the data revealed the following remarks: (i) The pre-exponential factor (collision factor; A) showed a direct relation to the activation energy E_a. (ii) The non spontaneity and endothermicity of the degradation processes. (iii) The perchlorato complex (**1**); $[Cu(L)(HL)(H_2O)_2]ClO_4.3H_2O$ which decomposes in one sudden step has abnormal higher values of A, E* and ΔH* [7]. Also, it has a unique positive ΔS* ~ 60 J mol^{-1} K^{-1} which is consistent with the formation of several species upon the sudden decomposition. (iv) In contrast, ΔS* has negative values for all the other complexes indicating that the activated species are more ordered than the reactants [17-19].

Biological activity

The isatinic hydrazone and its copper(II)- complexes (**1-6**) were screened for both antibacterial and antifungal activities (Table 5). Inspection of the data revealed that all complexes lack the activity towards the fungus; *Fusarium oxysporum*. Also, the ligand and its complexes (**1**, **4** and **5**) lack the antimicrobial and antifungal activities. On the other hand, only the chloro-complex (**3**) has high activity against Gram-positive bacteria (*S. aureus & S. pyogenes*) relative to the nitrato-complex (**2**). Also, the sulfato-complex (**6**) exhibited some activity towards Gram-negative bacteria (*P. phaseolicola & P. fluorescens*) as well as the Fungus; *Aspergillus fumigatus*. In general, the antibacterial results (Table 5) suggest low activity as compared to the standard references (control) and depend mainly on type of the coordinated anion in consistency with our previous studies [14,20].

Experimental

Materials

The chemicals used in this investigation were of the highest purity available (Merck, BDH, Aldrich and Fluka). They included copper(II)- nitrate, perchlorate, sulfate, chloride, bromide and acetate as well as *o*-toluidine, ethyl acetoacetate, phosphorus oxychloride, hydrazine hydrate and isatin. Organic solvents were reagent grade chemicals and were used without further purification.

Measurements

Microanalyses were carried out on a Perkin-Elmer 2400 CHN elemental analyzer. Thermal analyses (TG-DSC) were carried out on a Shimadzu-50 thermal analyzer. Electronic spectra were recorded on a Jasco V-550 UV/VIS spectrophotometer. IR spectra were recorded on a Bruker Vector 22 spectrometer using KBr pellets. ESR spectra were recorded on a Bruker Elexsys, E 500 operated at X-band frequency. Mass spectra were recorded either at 70 eV on a gas chromatographic GCMSQP 1000-EX Shimadzu mass spectrometer or Direct Inlet unit (DI-50) of Shimadzu GC/MS - QP5050A. ^1H NMR spectra were recorded as DMSO-d_6 solutions on a

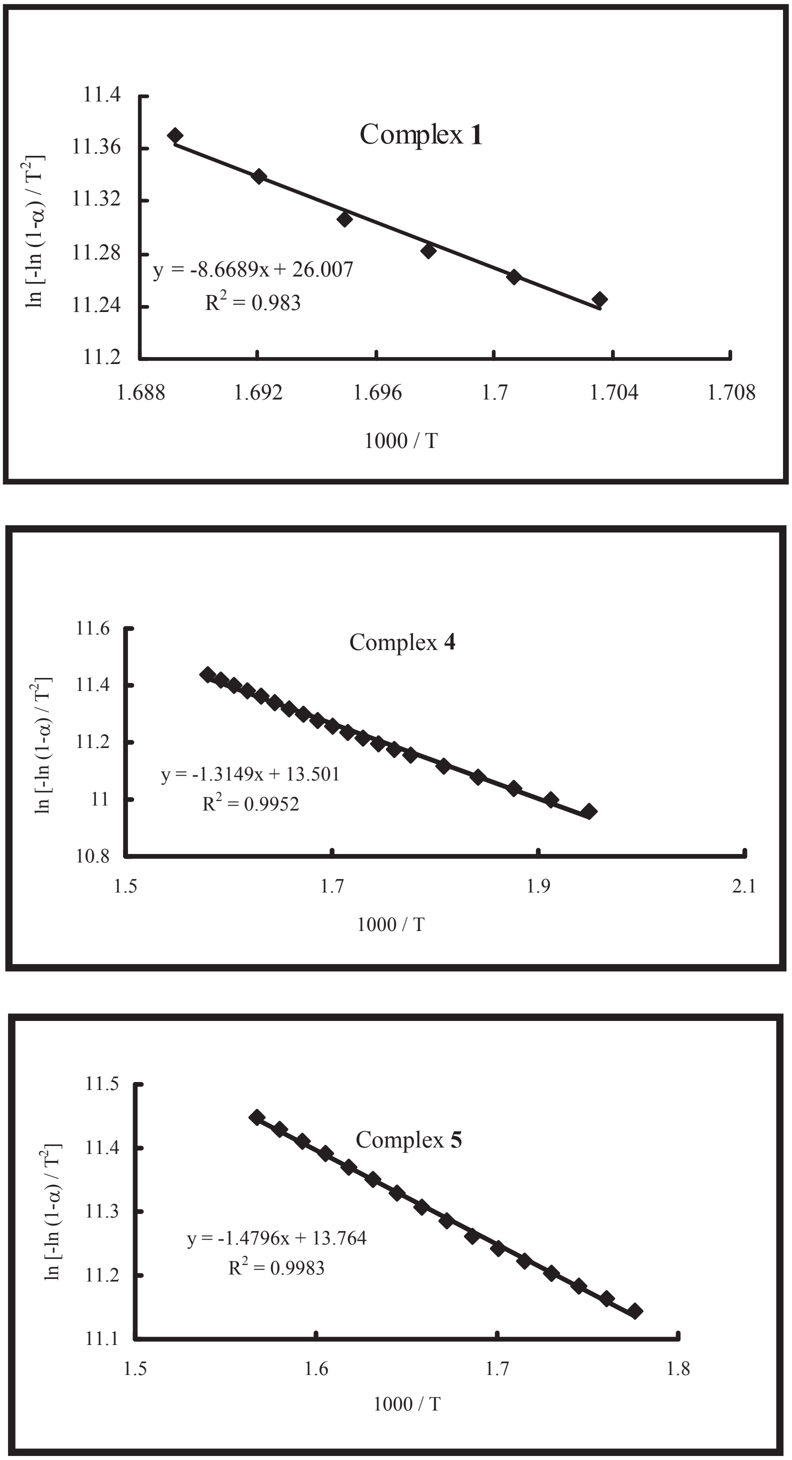

Figure 4 Evaluation of A and E* for the first stage of thermal decomposition.

Varian Mercury VX-300 NMR spectrometer using TMS as a reference. Molar conductivity was measured as DMF solutions on the Corning conductivity meter NY 14831 model 441. Magnetic susceptibility of the complexes was measured at room temperature using a Johnson Matthey, MKI magnetic susceptibility balance. Melting points were determined using a Stuart melting point apparatus.

Table 4 Thermodynamic and kinetic parameters[a] of the copper(II)- isatinic complexes.

Complex	Stage	T (K)	A × 10^{-9} sec^{-1}	E*	ΔH*	ΔG*	-ΔS*
1	1st	592	1.709 × 10^7	71.952	67.038	31.487	-60.053
2	1st	355	1.486	5.942	2.996	28.082	70.667
	2nd	475	5.627	9.123	5.18	34.644	62.029
4	1st	590	9.601	10.914	6.017	41.059	59.393
5	1st	602	14.053	12.281	7.284	41.235	56.398

[a] E*, ΔH* and ΔG* are in k J mol^{-1} while ΔS* is in J mol^{-1} K^{-1}.

Preparation of the hydrazone [9-11]

An ethanolic mixture of 2-hydrazinyl-4,8-dimethyl quinoline (0.01 mol) and isatin (0.012 mol) was refluxed for 1/2 h. The formed red compound was filtered off, washed with ethanol and crystallized from DMF. The results of elemental analysis, % yield and m.p°C are shown in Table 1.

Preparation of the metal complexes

Methanolic solutions of the metal salt and the ligand were mixed in the mole ratio 1:1 and/or 1:2; M:L and refluxed for 6-10 hours depending on the nature of the isolated complexes. The resulting precipitates were filtered off, washed with methanol then ether and finally air-dried. The results of elemental analysis, % yield and m.p°C are shown in Table 1.

Antimicrobial and antifungal activities

The standardized disc-agar diffusion method was followed to determine the activity of the synthesized compounds against the sensitive organisms *Staphylococcus aureus* (ATCC 25923) and *Streptococcus pyogenes* (ATCC 19615) as Gram-positive bacteria, *Pseudomonas fluorescens* (S 97) and *Pseudomonas Phaseolicola* (GSPB 2828) as Gram-negative bacteria and the Fungi *Fusarium oxysporum* and *Aspergillus fumigatus*. The antibiotic chloramphencol and Cephalothin were used as standard references in case of Gram-negative and Gram-positive bacteria, respectively, whereas Cycloheximide was used as a standard antifungal reference. The tested compounds were dissolved in DMF which have no inhibition activity to get concentration of 2 mg/mL. The test was performed on medium potato dextrose agar (PDA) which contain infusion of 200 g potatoes, 6 g dextrose and 15 g agar. Uniform size filter paper disks (3 disks per compound) were impregnated by equal volume (10 μL) from the specific concentration of dissolved tested compounds and carefully placed on inoculated agar surface. After incubation for 36 h at 37°C in case of bacteria and for three days at 25°C in case of fungi, inhibition of the organisms which evidenced by clear zone surround each disk was measured and used to calculate mean of inhibition zones.

Conclusion

The present study is planned to check the effect of the counteranions on the isolated complexes as well as the ligational behavior of the isatinic hydrazone ligand. The effect of anion for the same metal ion is obvious from either the geometry of the isolated complexes (O_h, T_d and D_{4h}) or the various modes of bonding. Also, the obtained complexes fulfill the strong coordinating ability of Cl^-, Br^-, NO_3^- and SO_4^{2-} anions as compared to the ClO_4^- anion in consistency with the donor ability of the anions [12]. In case of copper(II)- acetate, a unique homoleptic complex (**5**) was obtained in which the AcO^- anion acts as a base enough to quantitatively deprotonate the hydrazone. The isatinic hydrazone uses its lactim form in most complexes.

Authors' contributions

HSS conceived the study, participated in the sequence alignment and drafted the manuscript. GAE and BAE participated in the design of the study, analysis of the data and modification of the manuscript. HSS and MAM isolated and characterized the complexes. FIH prepared and characterized the organic ligand. All authors read and approved the final manuscript.

Competing interests

The authors declare that they have no competing interests.

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Table 5 The biological activity* of HL and its copper(II)- complexes.

Organisms	% inhibition				
	Gram - positive bacteria		Gram - negative bacteria		Fungus
	S. aureus	*S. pyogenes*	*P. Phaseolicola*	*P. fluorescens*	*Aspergillus fumigatus*
HL	–	–	–	–	–
(**2**)	7	–	–	–	–
(3)	24	22	–	–	–
(**6**)	–	–	9	22	18

*Complexes 1, 4 and 5 lack the biological activity.

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