Scientific paper

2-(Naphthalen-2-yl)-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde Oxime and Co(III) and Ni(II) Complexes of Tridentate Ligand Containing Oxime, Imine and Amine Donor Groups: Syntheses, Characterisation and Thermal Properties

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Received: 04-05-2012

Abstract

A novel 1,2,3,4-tetrahydroquinazoline oxime was synthesised from a condensation reaction of 2-(naphthalen-2-yl)-2-oxoacetaldehyde oxime with 2-aminobenzylamine. Subsequently, α-imine oxime complexes of this compound that formed with Co(III) and Ni(II) metal ions were obtained. All structures were characterised by spectral methods (FT-IR, NMR, AAS, mass), elemental analysis, thermal analyses, magnetic susceptibility and molar conductivity. The analyses confirmed the following molecular formulae: $[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$ and $[Ni(HL)_2]Cl_2$. The Co(III) complex is diamagnetic. Magnetic susceptibility measurements revealed octahedral geometry for the Co(III) and Ni(II) complexes. In the complexes, the chloride ion was found to be noncoordinated to the metal ions as confirmed by conductivity measurements. The NMR spectra of the Co(III) complex confirmed the presence ofethanol in the Co(III) complex. The experimental results of TG-DTA showed that the Co(III) complex contained ethanol and crystal water in the first two stages**.** The end of the thermal decomposition of the complexes yielded a final product of a metal oxide.

Keywords: 2-(Naphthalen-2-yl)-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime; Amine-imine-oxime; α-Imine oxime; Co(III) complex; Ni(II) complex

1. Introduction

Tetrahydroquinazolines have received considerable attention over the past years because of their wide range of biological activities and properties, such as the choleretic effect,[1] antibacterial,[2] antiinflammatory,[3] antiviral,[4] antimicrobial,[5] anticancer,[6,7] central nervous system(CNS) depressant,[8] and antioxidant.[9] Therefore, chemists have made many efforts to synthesise these compounds.[10–19] The formation of tetrahydroquinazoline is a synthesis of a Schiff base from aldehydes[10] and 2-aminobenzylamines; there is a tautomeric equilibrium between the tetrahydroquinazoline and the Schiff bases.[10–13] The chain-ring-chain tautomeric equilibrium depends on the substituent at position 2 (Figure 1).[11,12] In addition, the ring form can be easily identified by the presence of magnetically nonequivalent methylene protons.[12]

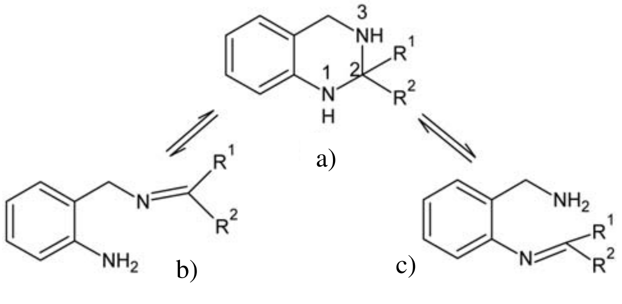


Figure 1. Tautomeric forms of the 1,3-unsubstituted tetrahydroquinazolines (a) Ring form, (b) aniline type chain form and (c) benzylamine type chain form.

Similarly, oxime compounds have been extensively studied because of their biological, industrial and structural importance.[20–22] For example, Tralkoxydim is a cyclohexanedione oxime derivative that is used as an agricultural herbicide.[23] Pralidoxime, obidoxime and methoxime are well known as antidotes for nerve agents.[24] Radicicol oxime derivatives also show significant antitumor activity.[25] Menthone oxime esters are perfumes and fragrant additives.[26] Dimethylglyoxime is used as a reagent to detect nickel.[27] Recently, only a few 1,2,3,4-tetrahydroquinazoline derivatives containing an oxime part that complexes with Ni(II) and Co(III) metal ions have been synthesised and characterised in detail.[28–31] Co(III) complexes of these ligands are similar in structure to those observed in the literature. During the formation of the Co(III) complexes, although there are different substituents at the 2-position in these ligands, the heterocyclic ring of the ligand was open, and an aniline-type chain form was observed.[29–31] As for the Ni(II) complex of 2-phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline, it was formed by the elimination of 1 mole of 2-aminobenzylamine from 2 moles of the ligand after a ring opening reaction by the Ni(II) attacks where ethanol acts as a reactant.[28]

In the present work, a newly synthesised 1,2,3,4-tetrahydroquinazoline oxime ligand,2-(naphthalen-2-yl)-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime, HL, which contains one oxime group and two -NH- groups, and amine-imine-oxime complexes resulting from the reactions with metal (II) salts has been prepared and identified by spectral (FT-IR, ^{1}H-NMR,^{13}C-NMR, AAS, mass) methods, elemental analyses, molar conductivity, magnetic susceptibility and thermal analysis (TG, DTA) techniques.

2. Experimental

2. 1. Materials and Measurements

2-(Naphthalen-2-yl)-2-oxoacetaldehyde oxime (INAN) was prepared according to a literature method[32] with little modification. All chemicals and solvents were purchased from Merck, Aldrich or Lachema and were used without further purification.

The ^{1}H–NMR and ^{13}C–NMR solution spectra were recorded at 25 °C on a Varian Mercury Plus 400 MHz spectrometer, utilising deuterated dimethylsulphoxide (DMSO–d_6) as a solvent. FT–IR spectra were recorded in the 4000–400 cm^{-1} region with a Thermo-Nicolet 6700 Fourier-Transform Infrared Spectrometer by using KBr pellets. Thermal analysis curves (TG and DTA) were obtained using a Seiko Exstar 6200 thermal analyser in a dry air atmosphere at a heating rate of 10 °C min^{-1} in the temperature range of 25–1000 °C using platinum crucibles. C, H and N microanalyses, metal analyses and mass spectra were carried out at the Technical and Scientific Research Council of Turkey, TUBITAK Bursa Test and Analysis Laboratory. Magnetic susceptibility measurements were performed at room temperature with a Sherwood Scientific MK1 model Magnetic Susceptibility Balance. Melting points were determined on a BÜCHI B–540 digital melting point apparatus and remained uncorrected. The molar conductivities of the complexes were measured by using a WTW model inoLab 730 conductivity meter.

2. 2. Preparation of 2-(Naphthalen-2-yl)-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime, (HL)

A solution of 2-aminobenzylamine (2-ABA) (10.0 mmol, 1.22 g) in 5 mL of absolute ethanol was added dropwise to a solution of INAN (10.0 mmol, 1.99 g) in 15 mL of absolute ethanol. The reaction mixture was stirred for 2 h at room temperature and left for one day at room temperature. The reaction product was filtered, washed with cold ethanol and finally dried under vacuum (Figure 2).

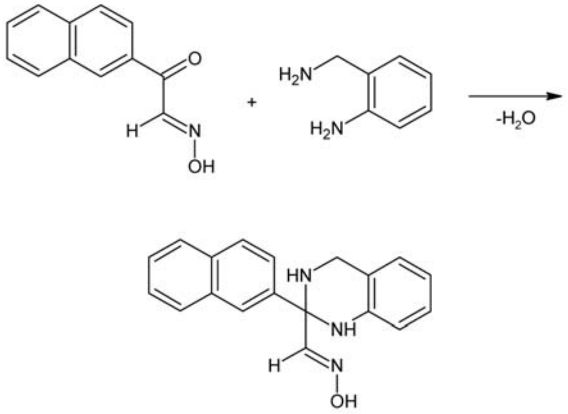


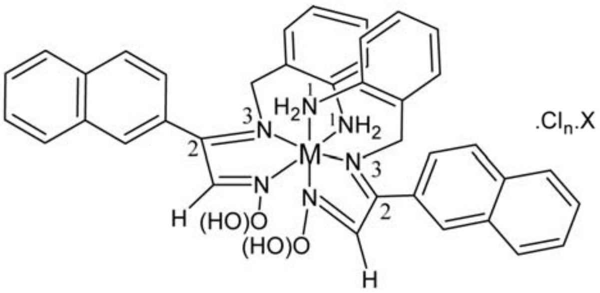
Figure 2. Synthesis of HL.

2. 3. Synthesis of [Co(L)$_2$]Cl · C_2H_5OH · 0,5 H_2O

A solution of $CoCl_2 \cdot 6H_2O$ (1.50 mmol, 0.366 g) in ethanol (10 mL) was added dropwise while stirring at room temperature to a solution of HL (3.00 mmol, 0.910 g) in ethanol (20 mL). Air-oxidation was achieved by stirring vigorously in air for 5 h. After 5 h, the precipitated orange-coloured compound was filtered, washed with cold ethanol several times, and dried under vacuum. The complex was named bis (1E,2E)-2-[(2-aminobenzyl)imino]-2-(naphthalen-2-yl)acetaldehyde oximato} cobalt(III) chloride monoethanol semihydrate (Figure 3).

2. 4. Synthesis of [Ni(HL)$_2$]Cl$_2$

A solution of $NiCl_2 \cdot 6H_2O$ (3.0 mmol, 0.71 g) in ethanol (10 mL) was added dropwise while stirring at room temperature to a solution of HL (3 mmol, 0.91 g) in ethanol (20 mL). The mixture was then stirred for 3 h at room temperature. After 3 h, the precipitated pinkcoloured compound was filtered and washed with cold ethanol several times, and dried. The complex was named bis{(1E,2E)-2-[(2-aminobenzyl)imino]-2-(naphthalen-2-yl)acetaldehyde oxime}nickel(II) chloride (Figure 3).



M = Co(III); n:1, X: $C_2H_5OH \cdot 0{,}5H_2O$ and –NO⁻
M = Ni(II); n:2 and –NOH

Figure 3. Suggested structures for the Co(III) and Ni(II) complexes.

3. Results and Discussion

The principal ligand HL was synthesised by the direct condensation of INANwith 2-ABA (Figure 2). The FT–IR spectrum of the solid form of HL and the NMR spectra of the HL in DMSO-d_6 solution indicate that the ligand remains in ring form. Regarding the FT–IR spectra spectra of Co(III) and Ni(II) complexes of HL, the stretching vibration band was determined to be caused by the –C=N stretching of the imine group. This finding shows that the Co(III) and Ni(II) complexes convert to chain form and coordinate to the metal ion in the chain form.

Two new complexes are readily formed by the reaction of the HL ligand with metal(II) chloride. It was determined that cobalt(II) is becoming oxidised to cobalt(III) in the $[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$ compound. The ligand HL undergoes deprotonation to form L^- in the Co(III) complex and acts as a tridentate ligand, thus occupying three positions in an octahedral geometry. The formula, colour, yield, molecular weight, melting point and elemental analysis results of the HL and Co(III) and Ni(II) complexes are shown below. Magnetic susceptibility, molar conductance and AAS results of the Co(III) and Ni(II) complexes are given in Table 1.

The elemental analyses and AAS results are in reasonably good agreement with the proposed structures. Room temperature magnetic susceptibility measurement of the mononuclear cobalt(III) complex reveals that the compound is diamagnetic, confirming the oxidation of cobalt(II) to cobalt(III) and structure's octahedral geometry.[30,31] The magnetic moment of the Ni(II) complex at room temperature has been determined to be 2.92 B.M., which is characteristic of a mononuclear octahedral Ni(II) complex. The molar conductance values, measured in methanol solutions (~1 × 10^{-3} M), for the Co(III) and Ni(II) complexes show that the Co(III) and Ni(II) complexes are 1:1 and 1:2 electrolytes, respectively.[33]

3. 1. LC–MS and FT–IR spectra

The mass spectrum of the free ligand that shows a molecular ion peak at $m/z = 303.8$ $(HL)^+$ indicates the formation of the desired structure. The peak at $m/z = 698.7$ at the mass spectrum of the cobalt(III) complex can be attributed to the $([Co(L)_2]Cl)^+$ ions.

The significant bands observed in the FT–IR spectra of the HL and its complexes are presented in Table 2. The FT–IR spectrum of the HL exhibited fairly strong bands at 3399–3265 cm^{-1} attributable to the ν(N–H) mode. The oxime (–OH) stretching vibration of the HL appeared as a broad peak maximum at 2766 cm^{-1}, which was assigned to the intramolecular hydrogen bonding vibration (N····H–O–). The bands observed at 1606 cm^{-1} and 939 cm^{-1} were attributed to stretching vibrations of the oxime (–C=N) and (N–O) groups, respectively. These values are in accord with those of previously reported 1,2,3,4-tetrahydroquinazoline oximes.[28, 30, 31] In addition, in the FT-IR spectrum of the α-carbonyl oxime derivative INAN, the (C=O) stretching frequency of the carbonyl group appeared at 1623 cm^{-1} but disappeared in the presence of the 1,2,3,4-tetrahydroquinazoline oxime, HL, demonstrating the formation of the new ligand.

In the FT–IR spectrum of the Co(III) complex, the ν(O-H) band due to water and ethanol molecules was as-

Table 1. Analytical and physical data of the ligand and its metal ion complexes.

Compound Molecular Formula	Colour	Yield (%)	Mol wt	mp (°C)	Found (Calculated) C	H	N	M	Λ_M (Ω^{-1} cm^2 mol^{-1})	μ_{eff} (BM)
HL	White	28	303.3	153	75.03	5.65	13.85	–	–	–
$C_{19}H_{17}N_3O$				dec	(75.22)	(5.65)	(13.86)			
$[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$	Orange	63	754.1	200	63.40	5.43	11.53	7.68	78	Diamagnetic
$C_{40}H_{39}N_6O_{3.5}CoCl$				dec	(63.70)	(5.21)	(11.15)	(7.90)		
$[Ni(HL)_2]Cl_2$	Pink	42	736.3	222	61.71	4.85	11.30	7.65	128	2.92
$C_{38}H_{34}N_6O_2NiCl_2$				dec	(61.98)	(4.65)	(11.42)	(7.97)		

Abbreviations: Mol wt, molecule weight; mp, melting point; dec, decomposition; Λ_M, molar conductance; μ_{eff}, effective magnetic moment.

signed at 3600–3200 cm^{-1}. This very broad band prohibits the appearance of other bands in the region. On the other hand, in the FT–IR spectrum of the Ni(II) complex, the bands at 3459 and 3202 cm^{-1} were associated with the (–NH_2) and (O–H) stretching vibrations, respectively. When the FT–IR spectra of the complexes are compared with those of the free ligand, the $\nu(C{=}N_{oxime})$ band is shifted to a lower frequency. This situation indicates that the oxime nitrogen must be coordinated to the metal ion. The (N–O) of the oxime group vibration in the Co(III) complex is situated at a frequency significantly higher (~98 cm^{-1}) than that of the free ligand, owing to the coordination of the N atom and the increase in bond order of the N–O bond upon deprotonation. A band around 1624–1638 cm^{-1} is attributable to the $(C{=}N_{imine})$ stretching vibration in the spectra of the Co(III) and Ni(II) complexes, which is absent in the spectra of the free ligand, indicating the coordination in the chain form. Additionally, the occurrence of new bands in the 478–470 cm^{-1} region in the FT–IR spectra of the metal complexes confirms the presence of metal-nitrogen bonds.

3. 2. ^{1}H– and ^{13}C–NMR Spectra

^{1}H–NMR spectra of the INAN, HL and Co(III) complex are summarised in Table 3. The ^{1}H–NMR spectrum of the ligand showed one peak as a multiplet around δ 3.24–3.20 ppm and two peaks as singlets at δ 6.79 ppm and δ 10.85 ppm, which disappeared upon addition of D_2O and were assigned to the NH proton adjacent to the –CH_2 group, the other NH proton and the proton of the oxime group, respectively. These signals are in agreement with values reported for similar compounds[28,30,31] and give evidence for 1,2,3,4-tetrahydroquinazoline ring formation. The resonance observed at δ 7.51 ppm as a singlet was assigned to the azomethine proton of the oxime group (HC=N). The signals of the proton of the aromatic groups are determined at ca. δ 8.02–6.45 ppm, and the –CH_2 group in the heterocyclic ring of the ligand resonated at δ 3.73 ppm and δ 3.46 ppm.

The ^{13}C–NMR spectrum of the INAN showed a signal for the carbon atom of the carbonyl group at δ 189.06 ppm while this was not observed in the ^{13}C–NMR spectra of the HL (Table 4). In addition, the ligand showed a signal corresponding to the quaternary carbon atom of the heterocyclic ring at δ 71.26 ppm, suggesting that the 1,2,3,4-tetrahydroquinazoline ring is formed.[28,30,31] The ^{13}C–NMR spectrum of the HL displayed a range of aromatic carbon resonances between δ 143.41–114.81 ppm. As for the carbon atoms of $H\underline{C}{=}NOH$ and –CH_2– groups in HL, they were observed at δ 152.60 ppm and δ 42.19 ppm, respectively.

A comparison of the ^{1}H–NMR and ^{13}C–NMR spectra of the ligand with those of the Co(III) complex confirms the coordination of the aniline-type chain form of the ligand. In the ^{1}H–NMR spectrum of the Co(III) complex, there is no OH peak belonging to the oxime group, providing evidence for its deprotonation. The chemical shift belonging to the –NH proton at position 3 in the HL disappeared from the ^{1}H–NMR spectrum of the Co(III) complex. After the complexation, in the integrated ^{1}H–NMR spectrum of the Co(III) complex, the observed proton resonances at ca. δ 6.61 ppm are due to the presence of the –NH_2 groups. The D_2O exchangeable peak was assigned by the formation of the aniline-type chain form in the Co(III) complex (Figure 3). In the Co(III) complex, the peaks due to the H–C=N group and methylene group of the HL showed an upfield shift and a downfield shift upon complexation with Co(III),[30,31] respectively. This finding confirmed that the nitrogen atoms of the oxime group and imine group were coordinated to the metal ion. In addition, in the ^{1}H–NMR spectrum of the Co(III) complex, the presence of proton resonances as a quartet at δ 3.40 ppm and as a triplet at δ 1.03 ppm due to the –CH_3 and –CH_2– groups of ethanol was observed. These signals proved the presence of ethanol in the Co(III)complex. At the same time, this conclusion is consistent with the results of the infrared spectrum and thermal analysis.

The chemical shift due to the quaternary carbon atom in the HL disappeared from the ^{13}C–NMR spectrum of the cobalt(III) complex. After complexation, a new resonance at δ 174.57 ppm was assigned through the formation of the aniline–type chain form (Figure 1). The chemical shifts belonging to the carbon atoms of the oxime group and methylene group in the HL showed an upfield shift and a downfield shift upon complexation with Co(III)[30] and appeared at δ 139.89 ppm and δ 53.77 ppm,

Table 2. Characteristic infrared bands (υ, cm^{-1}) of the INAN, HL and its metal ion complexes (KBr pellets).

Compound	**NH/ (NH_2)**	**O–H / H_2O (O–H–N)**	**$\underline{H–C}$=N**	**C=O**	**C=N**		**N–O**	**M–N**
					imine	**oxime**		
INAN	–	3336	2913	1623 s, sh	–	1597	993 m, sh	–
HL	3399 s, sh; 3265 s, sh	(2766)	2904	–	–	1606	939 m, sh	–
$[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$	–	3600–3200	2925	–	1624 w	1582 w	1037 w	478 w
$[Ni(HL)_2]Cl_2$	(3459)	3202	2917	–	1638 w	1595 w	948 w	470 w

Abbreviations: υ, stretchingvibration; w, weak; m, medium; s, strong; sh, sharp.

Table 3. ^{1}H–NMR spectral data (δ, ppm) and coupling constants (Hz) of the INAN, HL and its Co(III) complex. (in DMSO–d_6).

Compound	OH*	H–C=N	Naphthalene	Phenyl	NH_2	NH**	>CH_2	–CH_3
INAN	12.75 s, 1H	8.19 s, 1H	8.69 s, 1H; 8.09 d (J = 8), 1H; 8.03–7.96 m, 3H; 7.67 t, 1H; 7.61 t, 1H	–	–	–	–	–
HL	10.85 s, 1H 1H	7.51 s, 1H	8.02 s, 1H; 7.87–7.82 m, 3H; 7,74 dd (J = 8.8, 1.6), 1H; 7.49–7.44 m, 2H	6.93 dt (J = 7.6, 1.6), 1H; 6.80 d (J = 9.2), 1H; 6.71 d (J = 6.8), 1H 6.45 td (J = 7.4, 1.2), 1H	– –	6.79 s, 1H; 3.24–3.20 m, 1H	3.73 dd (J = 16.6, J = 0.4), 1H; 3.46 dd (J = 16.6, J = 0.4), 1H	
$[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0,5H_2O$	**	7.04 s, 2H	8.24–8.08 m, 8H; 7.75–7.66 m, 6H	7.32–7.16 m, 10H	6.61 (2H)	–	4.95 d (J = 14.0), 2H; 4.87 d (J=14.8), 4H; 3.40 q*	1.03 t*

* D_2O exchange, s: singlet, t: triplet, m: multiplet, q; quartet, b: broad, sh: sharp, *: ethanol peak, **: not observed

respectively. These results substantiate the conclusion obtained by the ^{1}H–NMR study and demonstrate the involvement of the oxime nitrogen and position of N–3 in coordination. Additionally, the ^{13}C–NMR spectrum of the Co(III) complex displayed a range for the aromatic carbon resonances between δ 139.14–123.96 ppm and a signal at δ 19.02 ppm due to the resonance of the carbon atom (–CH_3) of ethanol.

Consequently, the HL ligand is coordinated with the Co(III) and Ni(II) ions through the nitrogen atoms of the amine, imine and oxime groups.

3. 3. Thermal Analyses

Thermal behaviours of the ligand and its complexes have been investigated using thermal analysis techniques. The thermal stability data are listed in Table 5. The HL is stable up to 139 °C and then decomposes in three clearly separate temperature regions. Decomposition is completed at 638 °C (Figure 4). TG–DTA curves for the Co(III) complex are given in Figure 5. The Co(III) complex loses ethanol between 28–101 °C (found: 5.5 %, calcd.: 6.1 %) and crystal water between 101–165 °C (found: 1.8 %, calcd.: 1.2 % weight loss). Because these two processes overlap, the weight loss of both is in agreement with the calculated one. After exceeding this temperature, the organic ligands start to decompose. Decomposition occurs in the temperature range of 165–664 °C in three steps, as shown by exothermic DTA peaks at 192, 407 and 547 °C. The exothermic peak at 407 °C is low in intensity; the enthalpy of the second reaction is small.The mixture of Co_3O_4/CoO is caused by the removal of the organic ligands and halogen. The decomposition product Co_3O_4 is converted to CoO between 664 and 933 °C (DTA_{max}: 893 °C).[34] This finding demonstrates that the organic ligand acts as a reducing agent during thermal decomposition. The final decomposition product is CoO (found: 9.6 %, calcd.: 9.9 %).

The Ni(II) complex is thermally stable up to approximately 73 °C. The complex shows three exothermic stages in the decomposition process. These stages occur between 73–243 °C (DTA_{max}: 220 °C), 243–467 °C (DTA_{max}: 409 °C) and 467–587 °C (DTA_{max}: 534 °C) (Figure 6). The total mass loss of the decomposition steps amounts to 88.3 % (calcd.: 89.9 %) for the Ni(II) complex. It corresponds to the removal of the HL ligands and chlorides leaving NiO as a residue.

4. Conclusion

In the present work, 2-(naphthalen-2-yl)-1,2,3,4-tetrahydroquinazoline-2- carbaldehyde oxime, HL, and α-imine oxime complexes of this compound that formed with Co(III) and Ni(II) metal ions were synthesised and structurally characterised. The HL ligand acts as a triden-

Table 4. ^{13}C–NMR spectral data (δ, ppm) of the INAN, HL and its Co(III) complex (in DMSO–d_6).

	INAN	HL	$[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$
$\underline{C}$=O	189.06	–	–
R–$\underline{C}$=N	–	–	174.57
H–$\underline{C}$=N	148.12	152.60	139.89
$\underline{C}$–C$<^{N}_{N}$	–	143.41	–
$>\underline{C}<^{N}_{N}$	–	71.26	–
$(C)_{aromatic}$	135.45–125.21	141.32–114.81	139.14–123.96
$\underline{C}H_2$	–	42.19	53.77
$\underline{C}H_3$	–	–	19.02*

*: belongs to ethanol, R: naphthalene

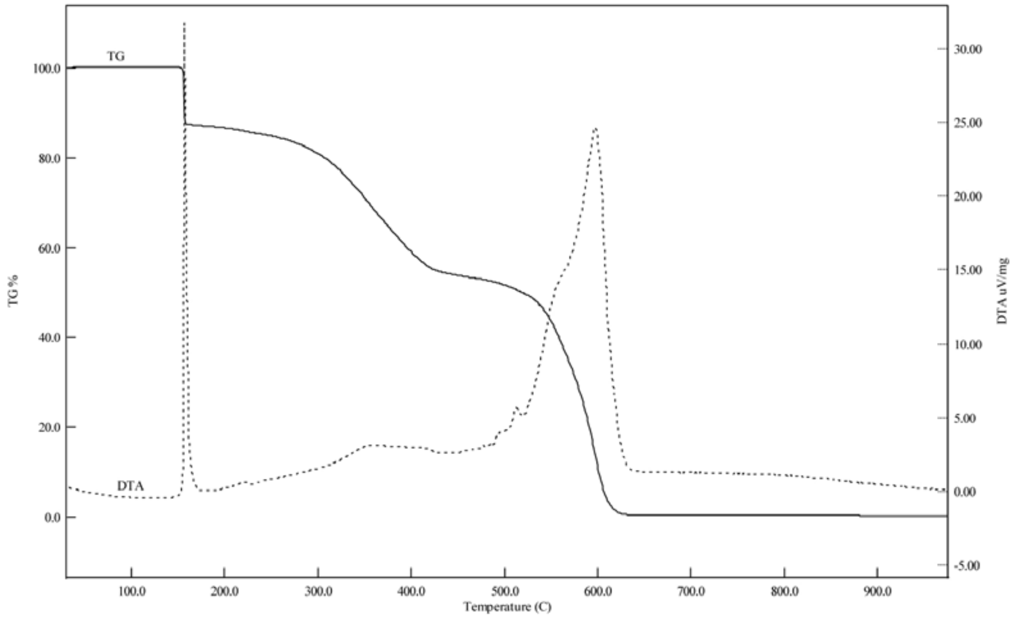


Figure 4. TG and DTA curves of HL (initial mass: 8.136 mg).

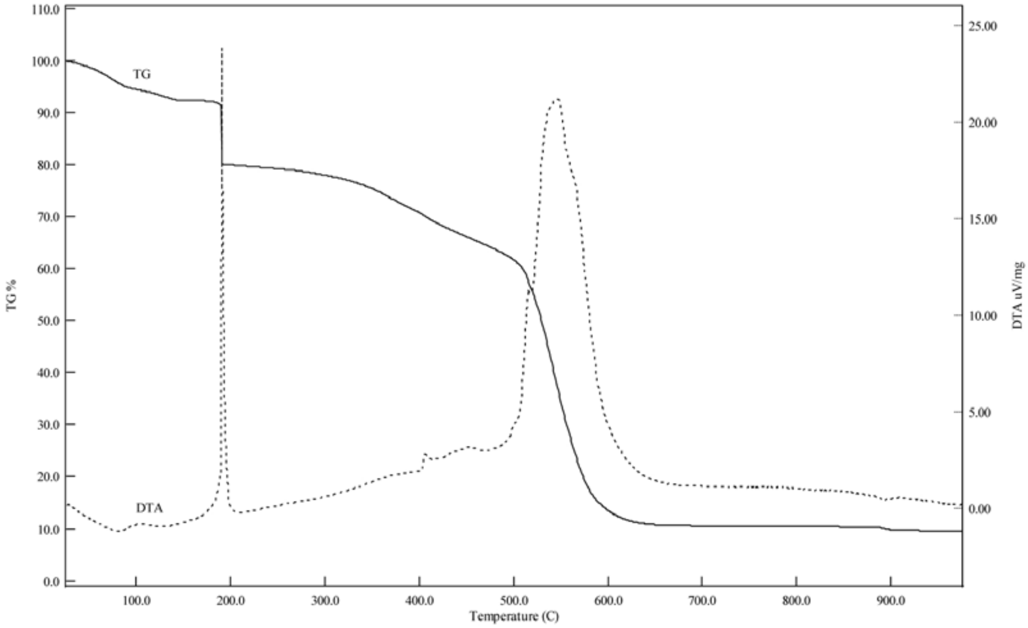


Figure 5. TG and DTA curves of $[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$ (initial mass: 6.905 mg).

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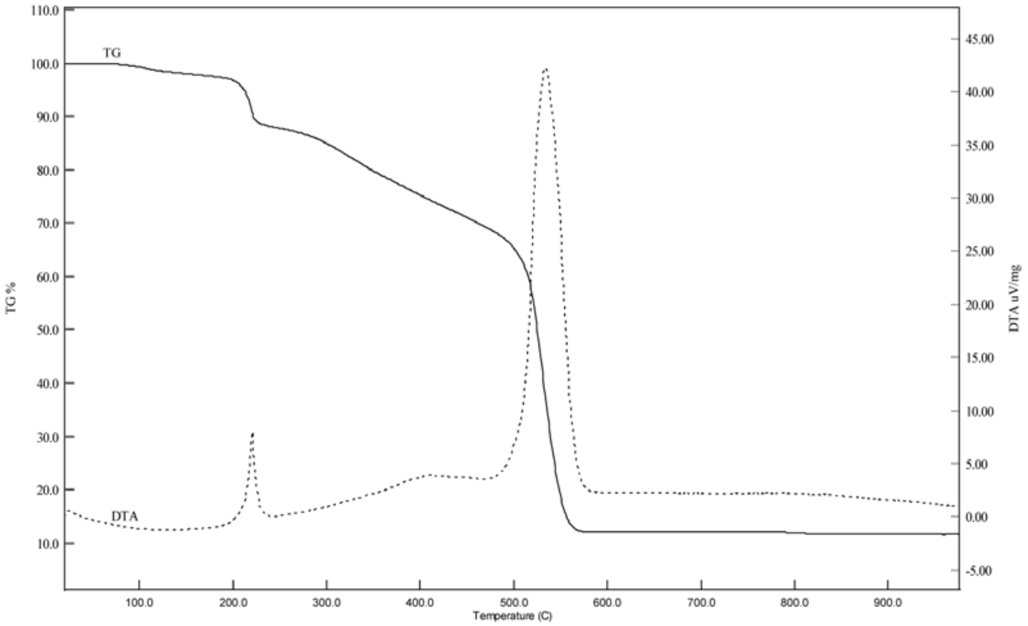

Figure 6. TG and DTA curves of $[Ni(HL)_2]Cl_2$ (initial mass: 5.027 mg).

Table 5. TG–DTA data of the HL and its metal ion complexes.

Compound	Temperature range, °C	DTA_{max}, °C	Total weight loss Found,%	Calculated, %	Assignment
HL	139 – 174	157(–)	12.9	*	**
	174 – 450	358(–)	46.2	*	**
	450 – 638	598(–)	100	100	–
$[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$	28 – 101	79(+)	5.5	6.1	$[Co(L)_2]Cl \cdot C_2H_5OH$
	101 – 165	129(+)	1.8	1.2	$[Co(L)_2]Cl$
	165 – 206	192(–)	20.1	*	**
	206 – 472	407(–)	35.6	*	**
	472 – 664	547(–)	89.3	*	Co_3O_4/CoO
	664 – 933	893(+)	90.4	90.1	CoO
$[Ni(HL)_2]Cl_2$	73 – 243	220(–)	12	*	**
	243 – 467	409(–)	30.6	*	**
	467 – 587	534(–)	88.3	89.9	NiO

(+): endothermic, (–): exothermic, * :not computed, **: unknown

tate ligand, thus occupying three positions in an octahedral geometry. The heterocyclic ring of the ligand was opened during the complexation and an aniline-type chain form occurred within the complexes. According to the results of all analyses, suggested structures for the Co(III) and Ni(II) complexes are shown in Figure 2. The structure of the cobalt(III) complex is similar to that observed in the literature.[29,31] It was determined that cobalt(II) is oxidised to cobalt(III) in $[Co(L)_2]Cl{\cdot}C_2H_5OH{\cdot}0{,}5H_2O$. The overall results conclude that the complexes consist of two amine-imine-oxime ligands coordinating through the amine, imine and oxime nitrogen groups. The net (+) charge was balanced by one and two noncoordinated chloride anions for $[Co(L)_2]Cl{\cdot}C_2H_5OH{\cdot}0{,}5H_2O$ and $[Ni(HL)_2]Cl_2$, respectively.

5. Acknowledgements

This work was supported by the Technical and Scientific Research Council of Turkey, TUBITAK (Project No. 106T371) and the Research Foundation of Uludağ University (Project No. 2006/66).

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Povzetek

Z reakcijo kondenzacije med 2-(naftalen-2-il)-2-oksoacetaldehid oksimom in 2-(aminometil)anilinom smo sintetizirali nov substituiran 1,2,3,4-tetrahidrokinazolin oksim. S to spojino kot ligandom smo pripravili komplekse s Co(III) in Ni(II) kovinskimi ioni. Dobljeni koordinacijski spojini smo okarakterizirali s spektroskopskimi metodami (FT-IR, NMR, AAS, MS), elementno analizo, merjenjem magnetne susceptibilnosti in prevodnosti ter termično analizo. Rezultati meritev so skladni s sestavo koordinacijskih spojin, ki jih predstavljata formuli $[Co(L)_2]Cl \cdot C_2H_5OH \cdot 0{,}5H_2O$ in $[Ni(HL)_2]Cl_2$. Kobaltov kompleks je diamagneten.Merjenje magnetne susceptibilnosti in prevodnosti potrjuje oktaedrično geometrijo obeh koordinacijskih spojin. Merjenje prevodnosti raztopin obeh kompleksov dokazuje, da v nobenem izmed obeh kompleksov kloridni ioni niso koordinirani na kovinski ion. Iz NMR spektra kobaltovega kompleksa lahko sklepamo na prisotnost etanola. Rezultati TG-DTA meritev kažejo izgubo etanola in vode v prvih dveh stopnjah termičnega razpada spojine Co(III), končni produkt razpada pa je v primeru obeh koordinacijskih spojin kovinski oksid.