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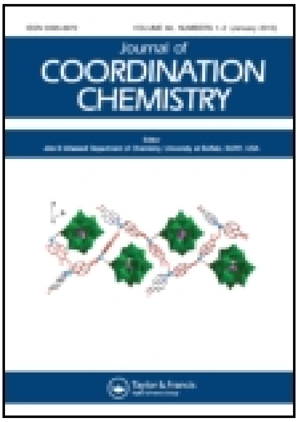

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Syntheses and characterizations of a novel 1,2,3,4-tetrahydroquinazoline and a Co(III) complex

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Syntheses and characterizations of a novel 1,2,3,4-tetrahydroquinazoline and a Co(III) complex

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The 2-furan-2-yl-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime (HL) and complex of tridentate ligand containing oxime, imine, and amine donors resulting from reactions with cobalt(II) salt have been synthesized and characterized by spectral methods (FT-IR, NMR, UV-Vis, mass), elemental analysis, magnetic susceptibility, molar conductivity, and thermal analysis (TG, DTA) techniques. The molecular structure of HL was elucidated from single-crystal X-ray diffraction. X-ray crystallographic analysis shows that the molecule crystallizes in the monoclinic system, space group *C*2/*c*. In the complex, chloride was not coordinated as confirmed by conductivity measurements. The metal is coordinated to the oxime, imine, and amine nitrogen atoms and decomposes with loss of the organic ligand and halogen to leave metal oxide. The metal to ligand ratio was 1 : 2 by elemental analysis and mass spectrum.

Keywords: 2-Furan-2-yl-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime; Amine-imine-oxime; Schiff base; Cobalt(III) complex; 2-Aminobenzyl amine

1. Introduction

The formation of Schiff bases by condensation of amines with aldehydes or ketones is well known [1–3]. Formation of 1,2,3,4-tetrahydroquinazolines is formally a synthesis of a Schiff base from aldehydes [4] or ketones and there is a ring-chain tautomeric equilibrium between the tetrahydroquinazolines and Schiff bases [4–7]. According to Sinkkonen and co-workers, 1,3-unsubstituted tetrahydroquinazolines have ring-chain tautomeric equilibria containing cyclic form, aniline type, and benzylamine type linear forms (scheme 1) [6]. The equilibrium depends on the substituents at position 2 in 1,2,3,4-tetrahydroquinazolines [5, 6].

The many and varied biological properties of quinazolines have led to their synthesis [4–13]. Some quinazolines have been identified as inhibitor of cyclin-dependent kinases by Sielecki and co-workers [14]. Many quinazoline derivative compounds exhibit biological activity [15].

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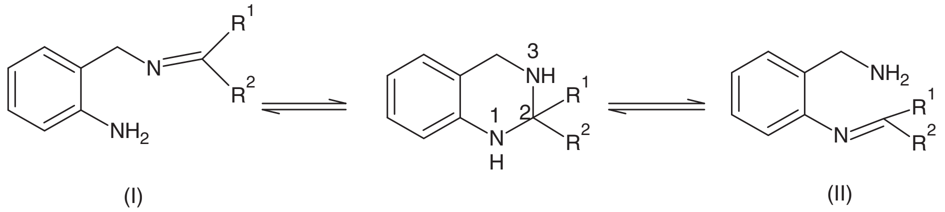

Scheme 1. Tautomeric forms of 1,3-unsubstituted tetrahydroquinazolines: (I) aniline type linear form; (II) benzylamine type linear form [6].

Oximes have been extensively studied because of their biological and structural importance [16–18], used as very efficient complexing agents for isolation, separation, and extraction of different metal ions [19–23]. Oximes were also reported for higher oxidation states of transition metals [24]. Imine-oxime ligands can stabilize higher metal oxidation states by π-donor character of the oxime and the negative charge on the oximato oxygen, partially neutralizing the positive charge on the metal [25–27]. Only a few 1,2,3,4-tetrahydroquinazoline derivatives, containing substituted oxime, have been synthesized, characterized, and used for complexation with some transition metal ions, i.e., Ni(II) [28] and Co(III) [29] complexes of 2-phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline and Co(III) complexes of 2-phenyl-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime and 2-thiophen-2-yl-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime [30].

In this study, 2-substituted 1,2,3,4-tetrahydroquinazoline, a new oxime, was synthesized from 2-aminobenzylamine and 2-(2-hydroxyimino-1-oxoethyl)furan and the amine-imine-oxime complex resulting from the reaction with cobalt(II) salt has been prepared and characterized by FT-IR, ^{1}H-NMR, ^{13}C-NMR, UV-Vis, mass spectral methods, elemental analysis, molar conductivity, magnetic susceptibility, and thermal analysis (TG, DTA). Determination of ligand structure was also performed by X-ray single-crystal analysis.

2. Experimental

2.1. *Materials and measurements*

2-(2-Hydroxyimino-1-oxoethyl)furan (INAF) was prepared according to literature method [21] with little modification. All chemicals and solvents were purchased from Merck, Aldrich, or Lachema and used without purification.

Melting points were determined on a BÜCHI B-540 digital melting point apparatus and are uncorrected. The molar conductivity of the complex was recorded using a WTW model inoLab 730 conductivity meter. Magnetic susceptibility measurements were performed at room temperature by using a Sherwood Scientific MK1 model Magnetic Susceptibility Balance. C, H, and N elemental analyses and mass spectra were carried out at the Technical and Scientific Research Council of Turkey, TUBITAK Bursa Test, and Analysis Laboratory. UV-Vis spectra were obtained in methanol solution ($10^{-4}\,\mathrm{mol\,L^{-1}}$ or $5 \times 10^{-5}\,\mathrm{mol\,L^{-1}}$) of the compounds with an ATI-Unicam UV2 spectrophotometer from 800 nm to 200 nm. ^{1}H-NMR and ^{13}C-NMR spectra were

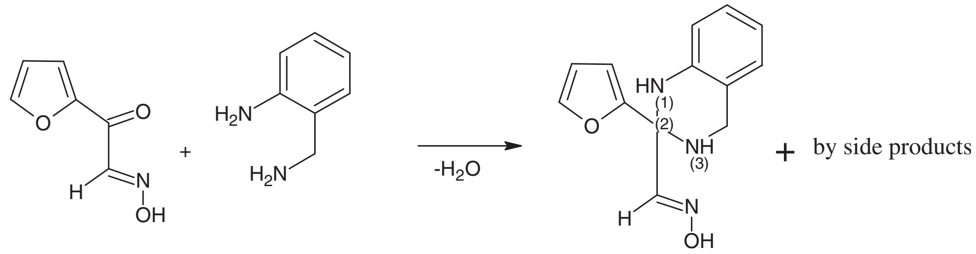

Figure 1. Synthesis of HL.

recorded at 25°C on a Varian Mercury Plus 400 MHz spectrometer, utilizing deuterated dimethylsulfoxide (DMSO-d_6) as a solvent. FT-IR spectra were recorded from 4000 cm^{-1} to 400 cm^{-1} with a Thermo-Nicolet 6700 Fourier-Transform Infrared Spectrometer by using KBr pellets. Thermal analysis curves (TGA and DTA) were obtained using a Seiko Exstar 6200 thermal analyzer in dry air at a heating rate of 10°C min^{-1} from 25°C to 1000°C using platinum crucibles.

2.2. *Crystallographic analyses*

Diffraction data for the ligand were collected with a Bruker AXS APEX [31] CCD diffractometer equipped with a rotation anode at 100 (2) K using graphite monochromated Mo-Kα radiation ($\lambda = 0.71069$ Å). Diffraction data were collected over the full sphere and were corrected for absorption. The data reduction was performed with the SAINT program package. For further crystal and data collection details see "Supplementary material." Structure solution was found with the SHELXS-97 [32] package using the direct methods and was refined by SHELXL-97 [33] against F^2. All non-hydrogen atoms were refined anisotropically. Hydrogen atoms were added to the structure model at calculated positions. Geometric calculations were performed with Platon [34]. Molecular drawings were obtained using Mercury [35].

2.3. *Synthesis of 2-furan-2-yl-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime (HL)*

A solution of 2-aminobenzylamine (10.0 m mol, 1.222 g) in absolute ethanol (10 mL) was added dropwise to a solution of 2-(2-hydroxyimino-1-oxoethyl)furan (10.0 m mol, 1.395 g) in absolute ethanol. The reaction mixture was stirred for 2 h at room temperature, left for a day at 4°C, and then kept 2 days at room temperature. The crystalline reaction product was filtered, washed with cold ethanol to remove the side products, and dried in air (figure 1). Yield (%): 23 (white crystals); m.p.: 121°C (decomposition point). Anal. Calcd for $C_{13}H_{13}N_3O_2$ (243.3 g mol^{-1}) (%): C, 64.2; H, 5.4; N, 17.3. Found: C, 63.6; H, 5.3; N, 17.7. FT-IR (KBr, ν cm^{-1}): N–H, 3391 s, 3270 s; O–H, 3196; H–C=N, 2970 w; >CH_2, 2881 w; C=N_{oxime}, 1605 s; N–O, 968 m. UV-Vis [methanol, λ_{max} nm (ε $10^4 (mol\,L^{-1})^{-1}\,cm^{-1}$)]: 208 (2.92); 238 (1.26); 295 (0.3). ^{1}H-NMR (DMSO-d_6, δ ppm): CH_2, 3.56 (d, $J = 16.4$, 1H); CH_2, 3.69 (d, $J = 16.8$, 1H); N_3H, 3.75 (s, 1H); H_{Het}, 6.32–6.31 (m, 1H); H_{Ar}, 6.38–6.37 (m, 1H); H_{Het}, 6.47

(d, $J = 3.2$, 1H); N_1H, 6.49 (s, 1H); H_{Ar}, 6.67 (d, $J = 8$, 1H); H_{Ar}, 6.76 (d, $J = 7.2$, 1H); H_{Ar}, 6.89 (t, $J = 8$, 1H); $^{H}{>}C{=}N$, 7.41 (s, 1H); H_{Het}, 7.60 (d, $J = 0.8$, 1H); –OH, 10.97 (s, 1H). ^{13}C-NMR (DMSO-d_6, δ ppm): $-CH_2$, 42.23; $^{Het}{>}C{<}$, 68.29; C_{Ar} or C_{Het}, 108.41, 110.66, 114.73, 116.60, 120.21, 125.88, 127.12, 143.16, 143.24, 150.28; H–C=N, 155.38. LC-MS (*m*/*z*): 243.8 $[HL + 1]^+$.

2.4. *Synthesis of bis{(1E,2Z)-[(2-aminobenzyl)imino](furan-2-yl)ethanal oximato}cobalt(III) chloride, [Co(L)$_2$]Cl*

A solution of HL (0.500 m mol, 0.122 g) in ethanol (15 mL) was added dropwise with stirring at room temperature to a solution of $CoCl_2 \cdot 6H_2O$ (0.25 m mol, 0.060 g) in ethanol (15 mL). Air-oxidation was achieved by stirring vigorously in air for 2 h. During the procedure an orange precipitate was formed after approximately 30 min. This complex was filtered and washed with water several times, and dried at room temperature. The complex is bis{(1E,2Z)-[(2-aminobenzyl)imino](furan-2-yl)ethanal oximato}cobalt(III) chloride (figure 2). Yield (%): 69 (orange precipitate); m.p.: 128–255°C (according to thermal analysis, decomposition range). Anal. Calcd for $C_{26}H_{24}N_6O_4ClCo$ (578.9 g mol^{-1}) (%): C, 53.9; H, 4.2; N, 14.5. Found: C, 53.5; H, 4.0; N, 15.1. FT-IR (KBr, ν cm^{-1}): N–H, 3397 s, 3207 s; C=N, 1594 s; N–O, 1028 m; Co–N, 535. UV-Vis [methanol, λ_{max} nm (ε 10^4 (mol L^{-1})$^{-1}$ cm^{-1})]: 203 (2.24), 294 (1.92), 355 (1.33), 420 (0.20). ^{1}H-NMR (DMSO-d_6, δ ppm): 2CH$_2$, 5.15 (d, $J = 14.8$, 2H); 2CH$_2$, 5.46 (d, $J = 14.8$, 2H); 2H$_{Ar}$, 6.41 (d, $J = 11.6$, 2H); H_{Het}, 6.86 (dd, $J = 3.6$, 2, 2H); 2H$_{Ar}$, 6.94 (d, $J = 7.2$, 2H); 2H$_{Ar}$ and 2NH$_2$, 7.16–7.10 (m, 6H); H–C=N, 7.36 (s, 2H); 2H$_{Ar}$, 7.40–7.38 (m, 2H); H_{Het}, 7.55 (d, $J = 4$, 2H); H_{Het}, 8.16 (d, $J = 1.2$, 2H). ^{13}C-NMR (DMSO-d_6, δ ppm): 2CH$_2$, 53.82; 2C$_{Ar}$ or 2C$_{Het}$, 112.98, 119.00, 123.08, 125.90, 128.71, 129.65, 131.62, 137.88, 139.01, 146.80; 2H–C=N, 146.34; 2Het–C=N, 159.43. LC-MS (*m*/*z*): 542.8 $[Co(L)_2]^+$; μ_{eff}: diamagnetic; molar conductance (methanol, Λ_M Ohm^{-1} cm^2 mol^{-1}): 82.

3. Results and discussion

3.1. *Crystal structure*

The molecular structure of HL with the atom labeling is shown in "Supplementary material." The crystal data and the refinement details of HL and the selected bond lengths and angles are given in "Supplementary material." X-ray crystallographic analysis shows the molecule crystallizes in the monoclinic system, space group *C*2/*c*. The bond angles and bond distances of furan ring are within normal ranges. The furan ring and 1,2,3,4-tetrahydroquinazoline ring are nearly perpendicular with an angle of 89.94°. The planarity of 1,2,3,4-tetrahydroquinazoline ring is lost with N(3) above the plane of 1,2,3,4-tetrahydroquinazoline ring.

In the crystal there are intermolecular hydrogen bonds O–H···N and X–H···π (Supplementary material). Intermolecular X–H···π interaction occurs between N(3)–H(3) and Cg(3) [Cg3: C(3)–C(4)–C(5)–C(6)–C(11)–C(12)].The H-ring centroid (H···Cg) distance is 2.34 Å and the γ angle is 8.29°. The N–H···Cg angle, 159.1°, is below the optimal value (180°) for the strongest NH···π interaction.

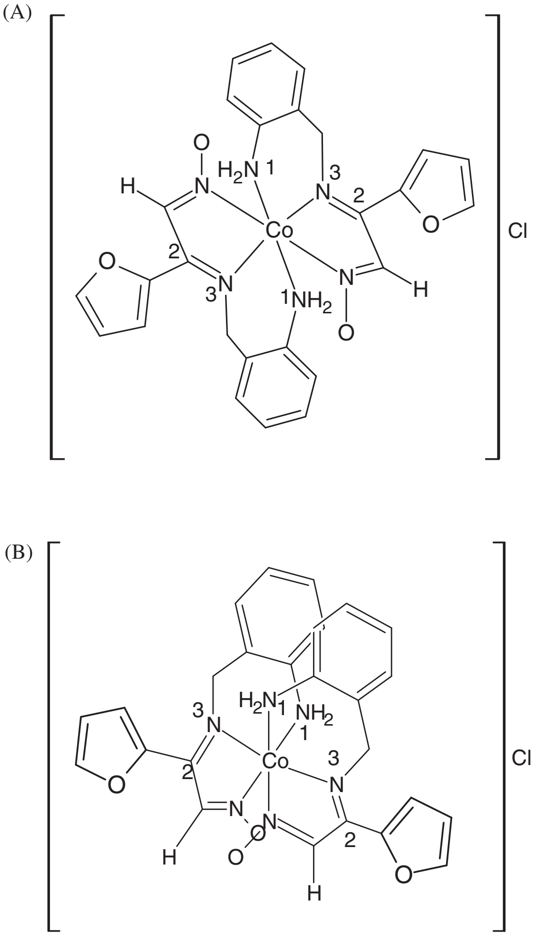

Figure 2. Suggested structural configurations for $[Co(L)_2]Cl$.

3.2. *^{1}H- and ^{13}C-NMR spectra*

In the ^{1}H-NMR spectrum of the HL, a singlet for –OH of oxime is observed at 10.97 ppm. The N–H proton adjacent to $-CH_2$ in the heterocyclic ring resonates at 3.75 ppm (1H as a singlet) and the other one resonates at 6.49 ppm (1H as a singlet). These signals are in agreement with values reported for similar compounds [5, 6, 8, 28, 30] and give evidence for 1,2,3,4-tetrahydroquinazoline ring formation.

The $-CH_2$ group in the heterocyclic ring of the ligand resonates at 3.56 ppm and 3.69 ppm as an AB system. The aldehyde proton adjacent to the oxime group resonates at 7.41 ppm. The ligand shows signals corresponding to the resonance of seven phenyl and furyl protons at *ca* δ 6.31–7.60 ppm.

The ^{13}C-NMR spectrum of HL shows a series of resonances, in accord with the number of carbon atoms of the ligand. In INAF, the signal of the carbon of carbonyl group appears at 175.28 ppm but disappears in the ^{13}C-NMR spectrum of the 2-substituted 1,2,3,4-tetrahydroquinazoline oxime, evidence for formation of HL.

In the ^{13}C-NMR spectrum of HL, signal at 42.23 ppm is attributed to methylene. The ligand shows signal corresponding to quaternary carbon at 68.29 ppm, evidence for tetrahydroquinazoline ring formation [28, 30]. There is only one signal due to the azomethine carbon atoms. This peak at 155.38 ppm belongs to $\underline{C}{=}N_{oxime}$ which proves that α-iminooxime is not formed. The NMR spectrum of HL agreed with those of known 2-substituted 1,2,3,4-tetrahydroquinazoline oximes [28,30]. All the signals from 108.41 to 150.28 ppm are assigned to carbon atoms of phenyl and furyl groups.

In the ^{1}H-NMR spectrum of the Co(III) complex, the signal due to –NOH is absent, evidence for deprotonation of oxime [36, 37]. The chemical shift belonging to –NH adjacent to $-CH_2$ in 2-substituted 1,2,3,4-tetrahydroquinazoline oxime disappeared from the ^{1}H-NMR spectrum of the cobalt complex. After complexation integration of the ^{1}H-NMR spectrum of the Co(III) complex indicates proton resonances at δ 7.14 ppm due to two $-NH_2$ protons of ligand. The downfield shift and D_2O exchangeability was assigned to the formation of the aniline type chain form. The ^{1}H-NMR signals of the $-NH_2$ protons of the ligand are downfield shifted compared to signals of the $-NH_2$ protons of free aniline. This is attributed to coordination of nitrogen at position 1 to cobalt in the complex (figure 2).

In the cobalt complex, the peak due to H–C=N showed considerable upfield shift, to δ 7.36 ppm, suggesting that the nitrogen of the oxime is a donor to the central metal. The phenyl and furyl protons are at δ 6.41–8.16 ppm.

In the complex, signals of methylene protons shift downfield to 5.46 and 5.15 ppm, confirming that nitrogen of $-C{=}N_{imine}$ coordinates [28–30].

The chemical shift is due to the quaternary carbon in HL disappearing from the ^{13}C-NMR spectrum of cobalt complex. However, after complexation a new resonance at δ 159.43 ppm was observed. Sinkkonen and co-workers determined the value of the chemical shift for carbon at position 2 of the aniline type chain form between δ 165.75 and 155.54 ppm [6]. In the ^{13}C-NMR spectrum of the Co(III) complex, a new resonance at δ 159.43 ppm shows that the heterocyclic ring of the ligand was opened during complexation and the aniline type chain form occurred in the Co(III) complex. Appearance of signal due to carbon of azomethine in α-iminooxime shifted downfield, evidence for nitrogen at position 3 of coordination [30].

The chemical shifts belonging to carbon atoms of oxime and methylene in HL showed an upfield shift and a downfield shift on complexation with Co(III) [30], appearing at δ 146.34 ppm and δ 53.82 ppm, respectively. These facts substantiate the conclusion obtained by the ^{1}H-NMR study, the involvement of the oxime nitrogen and position of N-3 in coordination.

3.3. *FT-IR spectra*

In HL the functional groups N–H, O–H, C=N, and N–O are readily identified from the infrared spectrum. The FT-IR spectrum of HL exhibited fairly strong bands at 3391–3270 cm^{-1} attributable to ν(N–H). Bands at 3196, 1605, and 968 cm^{-1} were attributed to the oxime ν(O–H), ν(C=N), and ν(N–O), respectively. These absorptions are in good agreement with known oximes and 1,2,3,4-tetrahydroquinazolines [5, 6, 8, 28, 30].

In the FT-IR spectrum of the complex, there is no peak for O–H since the complex forms with deprotonation of O–H. The peaks for $-NH_2$ were detected at 3397 and 3207 cm^{-1}. The C=N band of the complex shifts to lower wavenumber by 11 cm^{-1} compared to the free ligand, evidence of coordination through nitrogen of oxime. The N–O stretching vibration of the ligand shifts to higher wavenumber about 60 cm^{-1} for the cobalt (III) complex because of augmentation of the double bond character between N and O. The non-ligand band at 535 cm^{-1} is assigned to ν(M–N).

3.4. *LC-MS spectra*

An attempt has been made to determine the molecular mass of HL and its cobalt(III) complex by LC-mass spectral studies. The mass spectrum of the complex was also recorded to determine its stoichiometric composition. In HL, the molecular ion peak, $[HL+1]^+$, is observed at *m/z* 243.8. In the mass spectrum of the cobalt(III) complex, the only strong peak at *m/z* 542.8 can be related to $[Co(L)_2]^+$ ion. A second ion at a bit higher mass appears in the spectrum of the cobalt complex with quite low intensity in comparison with $[Co(L)_2]^+$. The peak at *m/z* 543.8 may be assigned to the negligible level of $[Co(HL)(L)]^+$.

3.5. *Molar conductance, magnetic susceptibility, and UV-Vis spectra*

The molar conductance of $[Co(L)_2]Cl$ was measured in methanol at 1×10^{-3} mol L^{-1} and was found to be 82 $Ohm^{-1} cm^2 mol^{-1}$. The molar conductance indicates 1 : 1 electrolytic of this compound [38]. The room temperature magnetic moment of the complex shows that it is diamagnetic. The electronic spectra of HL and its complex were measured in methanol at room temperature. HL exhibits three main absorptions at λ_{max} 208, 238, and 295 nm corresponding to intra-ligand transitions ($\pi-\pi^*$ and $n-\pi^*$). The electronic spectrum of $[Co(L)_2]Cl$ shows intense bands at 203, 294, and 355 nm, assigned to intra-ligand ($\pi-\pi^*$ and $n-\pi^*$) transitions associated with aromatic ring and C=N chromophores. Spectra of the complex showed intense absorption at 420 nm, which could be associated with ligand-to-metal charge transfer transitions.

Previously, we reported the synthesis of Co(III) complexes of 2-phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline [29], 2-phenyl-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime, and 2-thiophen-2-yl-1,2,3,4-tetrahydroquinazoline-2-carbaldehyde oxime [30]. In each of these complexes, ligands are deprotonated tridentate chelates and the cobalt(III) is octahedrally coordinated by six nitrogen atoms of two ligands. Only Co(III) complex of 2-phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline contains two different oximes. 2-Phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline is the deprotonated form in this complex and coordinates to cobalt(III), while the other stays free in the crystal structure [29]. Although there are different substituents at position 2 in these ligands, aniline type chain form is maintained in the cobalt complexes. According to Colak and co-workers, Ni(II) complex of 2-phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline was formed by elimination of 1 mole of 2-aminobenzylamine from the 2 moles of the ligand after ring opening attack by Ni(II) with ethanol as reactant [28].

In this study, the heterocyclic ring of the new ligand was opened during complexation and aniline type chain form occurred in the Co(III) complex. However, the effect of the opening of the ring on substituents at position 2 should be investigated in more detail.

3.6. *Thermal analyses*

HL exhibits three decomposition stages (Supplementary material). The ligand involves consecutive decompositions with exothermic effects (DTA_{max}: 124°C and 260°C) from 110°C to 407°C. The third stage is related to burning of the remaining organic residue, from 407°C to 619°C (DTA_{max}: 551°C).

Thermal decomposition of $[Co(L)_2]Cl$ proceeds in two exothermic stages between 128°C and 253°C (DTA_{max}: 230°C) and 253–673°C (DTA_{max}: 528°C), respectively (Supplementary material). The compound is thermally stable to 128°C and does not contain hydrated or lattice water molecules. The decomposition product Co_3O_4 is converted to CoO between 673°C and 953°C (DTA_{max}: 921°C). The weight loss of 89.3% suggests that the final product may be CoO (Found: 10.7, Calcd: 12.9).

4. Conclusion

HL and a cobalt(III) complex, $[Co(L)_2]Cl$, have been synthesized. The crystal structure of ligand exhibits intermolecular hydrogen bonds of the $O–H\cdots N$ and $X–H\cdots\pi$ interactions. The heterocyclic ring of the ligand was opened during complexation and aniline type chain form occurred in the complex. The complex consists of two amine-imine-oxime ligands coordinating through their amine, imine, and oxime nitrogen atoms and has octahedral geometry around cobalt(III) with a net 1+ charge balanced by one non-coordinated chloride. The oxime hydrogen is absent in the complex.

The structure of complex may be similar to figure 2(B), because the structure of the Co(III) complex of 2-phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline obtained from its crystalline form is similar to that [29] and the very low intensity peak in the mass spectrum of the complex may be the evidence for this coordination.

Supplementary material

Crystallographic data for the structure of HL reported in this article has been deposited with the Cambridge Crystallographic Data Centre as CCDC 812526. The data can be obtained free of charge *via* www.ccdc.cam.ac.uk/data_request/cif.

Acknowledgments

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