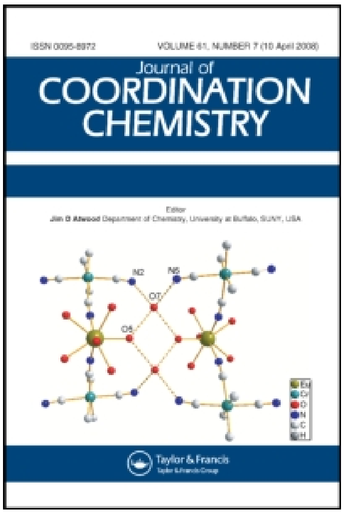

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# A Co(III) complex with a tridentate amine-imine-oxime ligand from 1,2,3,4-tetrahydroquinazoline: synthesis, crystal structure, spectroscopic and thermal characterization

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# A Co(III) complex with a tridentate amine-imine-oxime ligand from 1,2,3,4-tetrahydroquinazoline: synthesis, crystal structure, spectroscopic and thermal characterization

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A cobalt(III) complex $[Co(L)_2]Cl \cdot PPO \cdot H_2O$ (**1**) (HL = 1-(2-aminobenzylimino)-1-phenyl-propan-2-one oxime, PPO = 1-phenyl-1,2-propanedione-2-oxime) has been synthesized and characterized by elemental analyses, spectral, thermal, magnetic and molar conductance measurements and single crystal X-ray diffraction. It crystallizes in the monoclinic crystal system, space group $P2_1/c$. Complex **1** consists of one uncoordinated water, one 1-phenyl-1,2-propanedione-2-oxime molecule, one *bis*[1-(2-aminobenzylimino)-1-phenyl-propan-2-one oxime]cobalt(III) cation and one uncoordinated chloride. The coordination geometry around Co is slightly distorted octahedral, completed with six nitrogens of two $L^-$ ligands. The oxime moieties have E configurations. In the crystal structure, intramolecular O–H···Cl and N–H···O and intermolecular N–H···O, O–H···Cl and N–H···Cl hydrogen bonds link the molecules into chains parallel to the *c* axis; hydrogen-bonded PPO molecules fill the spaces between the chains and stabilization of the structure.

*Keywords*: Cobalt(III); 1-Phenyl-1,2-propanedione-2-oxime; 2-Aminobenzylamine; Amine-imine-oxime

## 1. Introduction

Oxime and dioxime derivatives are very important in chemical industry and medicine [1]. They have a broad pharmacological activity spectrum, encompassing antibacterial, antidepressant and antifungal activities [2–4]. Some oxime complexes also have anti-carcinogenic activities [1, 5, 6]. The oxime (–C=N–OH), potentially ambidentate with possible coordination through nitrogen and/or oxygen, is a functional group that has not been explored in crystal engineering. In the solid state, oximes are usually associated via O–H···N hydrogen bonds of 2.8 Å. Oxime groups possess stronger

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hydrogen-bonding capabilities than alcohols, phenols, and carboxylic acids [7]; intermolecular hydrogen bonds combine moderate strength and directionality [8] in linking molecules to form supramolecular structures and has received considerable attention with respect to directional noncovalent intermolecular interactions [9].

Only the synthesis of a few amine-imine-oxime complexes have been reported in the literature [10], Co(III) complexes of 1-phenyl-1,2-propanedione-2-oxime, (PPO) [11–14] and 2-aminobenzylamine (2ABA) [15].

However, to our surprise, we could not find reports of complexes of the amine-imine-oxime, synthesized from 1,2,3,4-tetrahydroquinazoline. Previously, we reported the synthesis of 2-phenyl-2-(1-hydroxyiminoethyl)-1,2,3,4-tetrahydroquinazoline with 1-phenyl-1,2-propanedione-2-oxime and 2-aminobenzylamine [16]. In this study, a cobalt(III) complex $[Co(L)_2]Cl \cdot PPO \cdot H_2O$ **1** (HL: 1-(2-aminobenzylimino)-1-phenyl-propan-2-one oxime, PPO: 1-phenyl-1,2-propanedione-2-oxime) has been synthesized and characterized by elemental analyses, spectral, thermal, magnetic and molar conductance measurements and single crystal X-ray diffraction.

## 2. Experimental

### 2.1. *Materials and instrumentation*

All chemicals and solvents were of reagent grade. $CoCl_2 \cdot 6H_2O$, 1-phenyl-1, 2-propanedione-2-oxime, 2-aminobenzylamine, $C_2H_5OH$ (Aldrich) were used as received. Elemental analyses for C, H and N were carried out using a Vario EL II CHNS Elemental Analyzer. Magnetic susceptibility measurements at room temperature were performed using a Sherwood Scientific MK1 model Gouy magnetic balance. UV–vis spectra were obtained on methanol solutions ($10^{-3}\,\mathrm{mol\,L^{-1}}$) of the complex with a Shimadzu Pharmaspec UV-1700 spectrometer in the range 1000–190 nm. FT–IR spectra were recorded from 4000–400 $\mathrm{cm^{-1}}$ with a Bruker Optics, Vertex 70 FT–IR spectrometer using KBr pellets. Diamond TG/DTA thermal analyzer was used to record simultaneous TG and DTG curves in nitrogen and DTA curve in static air at a heating rate of $10\,\mathrm{K\,min^{-1}}$ from 20–1000°C using platinum crucibles. The molar conductivities ($\Lambda_M$) were recorded using a WTW model 315i conductivity meter.

### 2.2. *Crystallographic analyses*

Experimental data are listed in table 1, selected bond lengths and angles and hydrogen-bond geometry are given in tables 2 and 3, respectively. Crystallographic data were recorded on an Enraf-Nonius CAD-4 diffractometer using Mo-K$\alpha$ radiation ($\lambda = 0.71073$ Å) at $T = 298$ K. Absorption correction by psi-scan [17] was applied. The structure was solved by direct methods [18] and refined by full-matrix least-squares against $F^2$ using all data [18]. All non-H atoms were refined anisotropically. Aromatic, methylene and methyl H atoms were positioned geometrically at distances of 0.93 (CH), 0.97 ($CH_2$) and 0.96 Å ($CH_3$) from the parent C atoms; a riding model was used during the refinement process and the $U_{iso}$(H) values were constrained to be $1.2U_{eq}$(carrier atom) for aromatic and methylene H and $1.5U_{eq}$(carrier atom) for methyl hydrogens. Other hydrogens were located in difference syntheses and refined

Table 1. Experimental data for **1**.

| Empirical formula | $C_{32}H_{32}N_6O_2Co \cdot C_9H_6NO_2 \cdot H_2O \cdot Cl$ |
|---|---|
| Color/shape | Purple/block |
| Formula weight | 808.20 |
| Temperature (K) | 298(2) |
| Radiation used, graphite monochromator | Mo-K$\alpha$ ($\lambda = 0.71073$ Å) |
| Crystal system | *P2₁/c* |
| Space group | Monoclinic |
| Units of cell dimensions (Å, °) | |
| $a$ | 10.0318(1) |
| $b$ | 30.7857(2) |
| $c$ | 13.8074(3) |
| $\alpha$ | 90.00 |
| $\beta$ | 111.14(3) |
| $\gamma$ | 90.00 |
| $V$ (Å$^3$) | 3977.3(8) |
| $Z$ | 4 |
| Absorption coefficient (mm$^{-1}$) | 5.5 |
| $D_{Calcd}$ (Mg m$^{-3}$) | 1.350 |
| Max. crystal dimen. (mm$^3$) | $0.15 \times 0.20 \times 0.30$ |
| $\theta$ (max) (°) | 26.30 |
| Reflections measured | 8028 |
| Range of $h$, $k$, $l$ | $-12 < h < 0$; $-38 < k < 0$; $-16 < l < 17$ |
| Diffractometer/scan | Enraf-Nonius CAD-4/$w$-$2\theta$ |
| Number of reflections with $I > 2\sigma(I)$ | 4554 |
| Corrections applied | Lorentz-polarization |
| Computer programs | SHELXS-97 [18] SHELXL-97 [18] ORTEP-3 [26] |
| Source of atomic scattering factor | Int. Table for X-ray Cryst. Vol. IV, 1974 [27] |
| Structure solution | Direct methods |
| Treatment of hydrogen atoms | Geometric calculation & difference map |
| No. of parameters var. | 527 |
| GOF | 1.03 |
| $R = \lVert F_o \rvert - \lvert F_c \rVert / \lvert F_o \rvert$ | 0.051 |
| $R_w$ | 0.142 |
| $(\Delta/\rho)$ max. (e A$^{-3}$) | 0.50 |
| $(\Delta/\rho)$ min. (e A$^{-3}$) | −0.33 |

isotropically [O–H = 0.92(5) and 0.95(7) Å, $U_{iso}$(H) = 0.015(3) and 0.015(3) Å$^2$ (for $H_2O$); O–H = 0.83(4) Å, $U_{iso}$(H) = 0.050 (15) Å$^2$ (for OH), and N–H = 0.87(4)–0.92(4) Å, $U_{iso}$(H) = 0.029(11)–0.046(13) Å$^2$ (for $NH_2$)].

### 2.3. *Synthesis of 1*

The synthesis of HL was achieved employing the literature method [16]. A solution of HL (0.53 g, 2 mmol) in ethanol-water (1 : 1, 20 mL) was added dropwise with stirring at 60°C to a solution of $CoCl_2 \cdot 6H_2O$ (0.24 g, 1 mmol) in ethanol (20 mL). The mixture was stirred for 4 h at 60°C and then slowly cooled to room temperature (scheme 1). The crystals formed were filtered, washed with 10 mL of cold ethanol and dried in air. Yield 77% (red crystals); $C_{41}H_{41}ClN_7O_4Co$ (790.20 g mol$^{-1}$); C, 62.8 (Calcd 62.32); H, 5.54 (5.23); N, 12.11 (12.41)%.

$^1$H NMR (DMSO-d$_6$) $\delta = 1.54$ (s, 4H, $-2 \times CH_2$), 1.98 (s, 3H, $-COCH_3$), 2.49 (s, 6H, $-C{=}NCH_3$), 3.98 (d, 2H, $J = 6.4$ Hz, $-CNH_2$), 4.67 (s, 2H, $-CNH_2$ bonding hydrogen with water), 6.31 (d, 2H, $J = 11.6$ Hz, $-H_2O$), 6.73 (d, 2H, $J = 11.2$ Hz, Ar–H),

Table 2. Selected bond lengths (Å), angles (°) and torsion angles (°).

| | | | |
|---|---|---|---|
| Co–N1 | 1.917(3) | N2–C32 | 1.323(4) |
| Co–N2 | 1.908(3) | N3–C1 | 1.293(4) |
| Co–N3 | 1.891(3) | N7–O4 | 1.375(5) |
| Co–N4 | 1.895(3) | N7–C40 | 1.268(5) |
| Co–N5 | 2.006(3) | C31–C28 | 1.438(5) |
| Co–N6 | 2.022(3) | C32–C1 | 1.435(5) |
| O1–N2 | 1.290(4) | C32–C30 | 1.494(5) |
| O2–N1 | 1.267(4) | C39–C40 | 1.487(7) |
| N1–C31 | 1.321(4) | C40–C41 | 1.487(7) |
| N1–Co–N2 | 87.14(12) | N3–Co–N6 | 94.08(14) |
| N1–Co–N3 | 92.71(13) | N4–Co–N5 | 92.36(14) |
| N1–Co–N4 | 82.53(13) | N4–Co–N6 | 90.42(15) |
| N1–Co–N5 | 89.19(13) | N5–Co–N6 | 96.25(14) |
| N1–Co–N6 | 171.28(15) | O2–N1–C31 | 122.2(3) |
| N2–Co–N3 | 82.14(13) | O1–N2–C32 | 122.4(3) |
| N2–Co–N4 | 94.95(13) | O4–N7–C40 | 112.7(4) |
| N2–Co–N5 | 171.32(14) | N1–C31–C28 | 112.3(3) |
| N2–Co–N6 | 88.37(13) | N2–C32–C1 | 112.3(3) |
| N3–Co–N4 | 174.57(13) | N7–C40–C39 | 113.6(4) |
| N3–Co–N5 | 90.19(14) | O2–N1–C31–C28 | −178.7(3) |
| O1–N2–C32–C1 | −175.8(3) | O4–N7–C40–C39 | −175.3(4) |

Table 3. Hydrogen-bond geometry (Å, °).

| D–H···A | D–H | H···A | D···A | D-H···A |
|---|---|---|---|---|
| O4–H4A···Cl | 0.83(4) | 2.21(5) | 3.036(4) | 174(4) |
| N6–H6A···O5 | 0.87(4) | 2.08(4) | 2.926(6) | 163(4) |
| N5–H51···O1$^{i}$ | 0.87(4) | 2.17(4) | 2.954(4) | 150(4) |
| O5–H5B···Cl$^{i}$ | 0.95(7) | 2.21(8) | 3.142(7) | 172(6) |
| N5–H52···Cl$^{ii}$ | 0.92(4) | 2.37(4) | 3.250(4) | 162(3) |

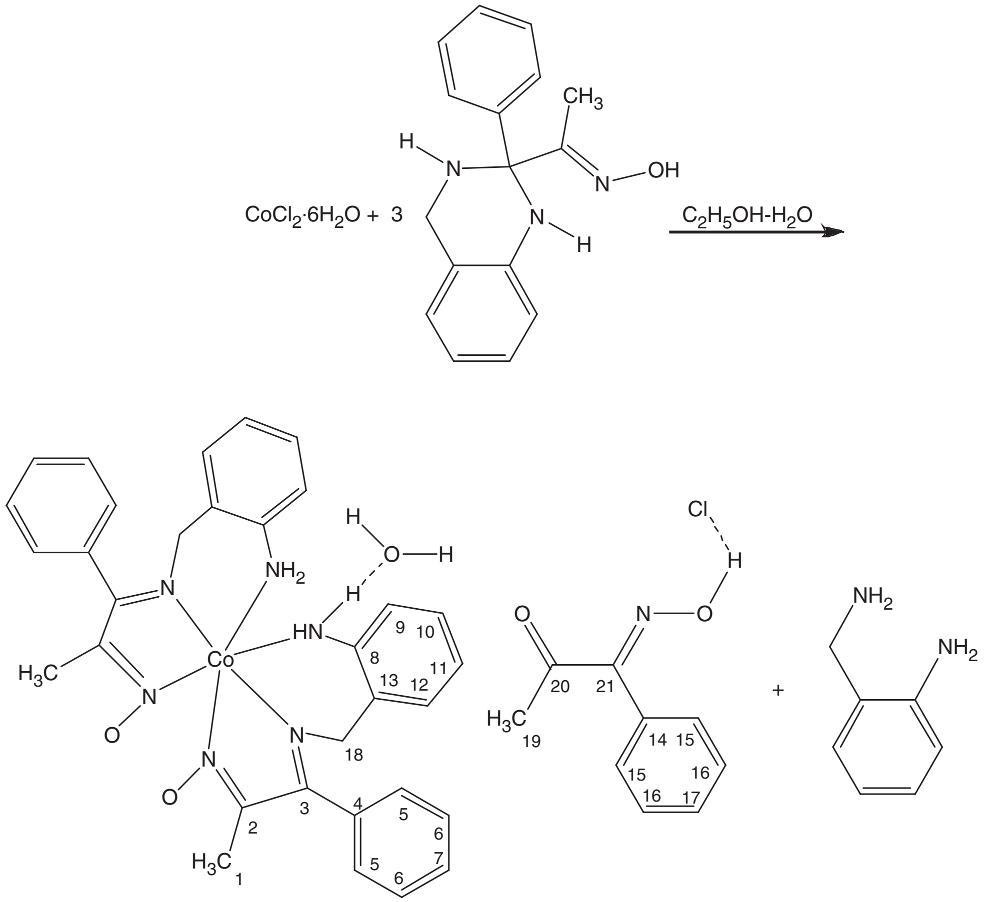
Symmetry codes: (i) $x$, $1/2-y$, $z-1/2$; (ii) $x$, $y$, $z-1$.

6.96 (t, 2H, $J = 8.8$ Hz, Ar–H), 7.06–7.09 (m, 2H, Ar–H), 7.11–7.16 (m, 5H, Ar–H), 7.40 (s, 1H, Ar–H), 7.44 (t, 3H, $J = 15.2$ Hz, Ar–H), 7.54–7.58 (m, 1H, Ar–H), 7.64 (s, 1H, Ar–H), 7.66 (s, 2H, Ar–H), 7.68–7.71 (m, 2H, Ar–H), 7.73–7.74 (m, 1H, Ar–H), 7.75–7.77 (m, 1H, Ar–H), 12.43 (s, 1H, CNOH). $^{13}$C NMR ([D$_6$]DMSO) $\delta = 10.24$ (C-18), 13.91 (C-1), 54.31 (C-19), 122.84, 126.00, 127.75, 128.37, 128.70, 128.76, 129.47, 130.45, 130.49, 131.48, 132.68, 133.23, 137.43, 139.58 (C-4–C-17; Ar–C), 146.79 (C-2), 155.51 (C-3), 176.10 (C-21), 192.54 (C-20).

## 3. Results and discussion

### 3.1. *IR spectra*

In IR spectra (table 4), the absorptions at 3318 and 3234 cm$^{-1}$ are due to $\nu(H_2O)$ vibrations of the crystal water and the characteristic OH$_{oxime}$ absorption [19] from PPO. The peaks at 3054 and 3021 cm$^{-1}$ are attributed to $\nu(NH_2)$ vibrations of HL.



Scheme 1. Synthesis of the cobalt(III) complex.

Table 4. IR spectral data (cm$^{-1}$).

| Assignment | **1** |
|---|---|
| $\nu OH_{water}$ | 3318 m |
| $\nu(NH_2)$ | 3054 m, 3021 m |
| $\nu(OH)_{oxime}$ | 3234 s |
| $\nu$(C-H) | 2892 w |
| $\nu(CH_2)$ | 2836 m |
| $\nu$(C=O) | 1657 vs |
| $\nu(C{=}N)_{imin}$ | 1619 s |
| $\nu(C{=}N)_{oxime}$ | 1593 m |
| $\nu$(C–N) | 1288 s |
| $\nu$(N–O) | 1017 s |
| Co–N | 443 w |

w – weak; m – medium; s – strong; vs – very strong; b – broad.

The peaks at 2892 and 2836 $cm^{-1}$ are due to $\nu(CH)$ vibrations of benzene rings and $\nu(CH_2)$ vibrations of HL. The peaks at 1657, 1619 and 1593 $cm^{-1}$ are due to $\nu(C{=}O)$ of PPO, $\nu(C{=}N_{imine})$ and $\nu(C{=}N_{oxime})$ vibrations. The peaks at 1288, 1069 and 1017 $cm^{-1}$ are assigned to $\nu(C–N)$ and $\nu(N–O)$ of HL ligand. The peak at 443 $cm^{-1}$ is due to Co-N stretch. Similar spectral results were also obtained for similar compounds [19–21].

### 3.2. *Electronic spectrum, magnetic study and molar conductance*

The d-d transition spectrum of the title complex in MeOH is compatible with a distorted octahedral configuration. The absorption spectrum of **1** has a band around 585 nm ($\varepsilon = 43.1\,cm^{-1}\,M^{-1}$), possibly due to $^1A_{1g} \rightarrow {}^1T_{1g}$ transition from a distorted octahedral stereochemistry. The room temperature magnetic moments of **1** show that it is diamagnetic. Molar conductivities were measured in DMF (dimethyl formamide) at $10^{-3}$ M. The molar conductance ($\Lambda_M$) was found to be 39 $\Omega^{-1}\,cm^2\,mol^{-1}$, indicating the ionic nature of the 1 : 1 electrolyte [22].

### 3.3. *Thermal analysis*

The DTA-TG-DTG curves of **1** are shown in figure 1. The first endothermic stage between 30 and 140°C ($DTG_{max} = 130$°C) corresponds to the loss of solvate water (found 2.4, Calcd 2.2%). The endothermic decomposition of L, $Cl^-$ and PPO molecule are observed in the subsequent stages between 140–637°C with $DTG_{max}$ peaks at 180, 200, 240, 330°C (found 90.3, Calcd 90.5%). The decomposition product $Co_3O_4$ is converted to CoO. The overall weight loss of 89.14% (Calcd 90.49%) agrees well with the proposed structure. Finally, the weight loss of 92.5% suggests that the residue may be CoO (found 7.5%, Calcd 9.3%).

### 3.4. *Crystal structure*

The X-ray structural determination of **1** confirms the structure assigned from spectroscopic data. The molecular structure of **1** along with the atom-numbering scheme is depicted in figure 2. The asymmetric unit contains one uncoordinated water, one 1-phenyl-1,2-propanedione-2-oxime, one *bis*[1-(2-aminobenzylimino)-1-phenyl-propan-2-one oxime]cobalt(III) cation and one uncoordinated chloride. Each $L^-$ is tridentate chelate and the cobalt is octahedrally coordinated by six nitrogens of two ligands. The PPO is not involved in coordination and remains as a solvate in the crystal structure. The Co–N bonds range from 1.891(3) to 2.022(3) Å, while the N–Co–N angles are in the range 82.14(13)–174.57(13)°, indicating distortion around cobalt(III) (table 2). The rings A (C2–C7), B (C8–C13), C (C14–C19), D (C20–C25), E (C33–C38), F (Co/N1/N4/C28/C31) and G (Co/N2/N3/C1/C32) are planar and the dihedral angles between B/C and F/G are 3.29(14)° and 83.89(8)°, thus rings B and C are nearly parallel, while F and G are nearly perpendicular to each other. On the other hand, rings H (Co/N4/N6/C14/C19/C27) and I (Co/N3/N5/C8/C13/C26) are not planar, having total puckering amplitudes, $Q_T$, of 0.734(4) and 0.800(5) Å, respectively, and distorted half-chair conformations $\phi = -43.3(4)$°, $\theta = 71.8(3)$° and $\phi = -46.1(3)$°,

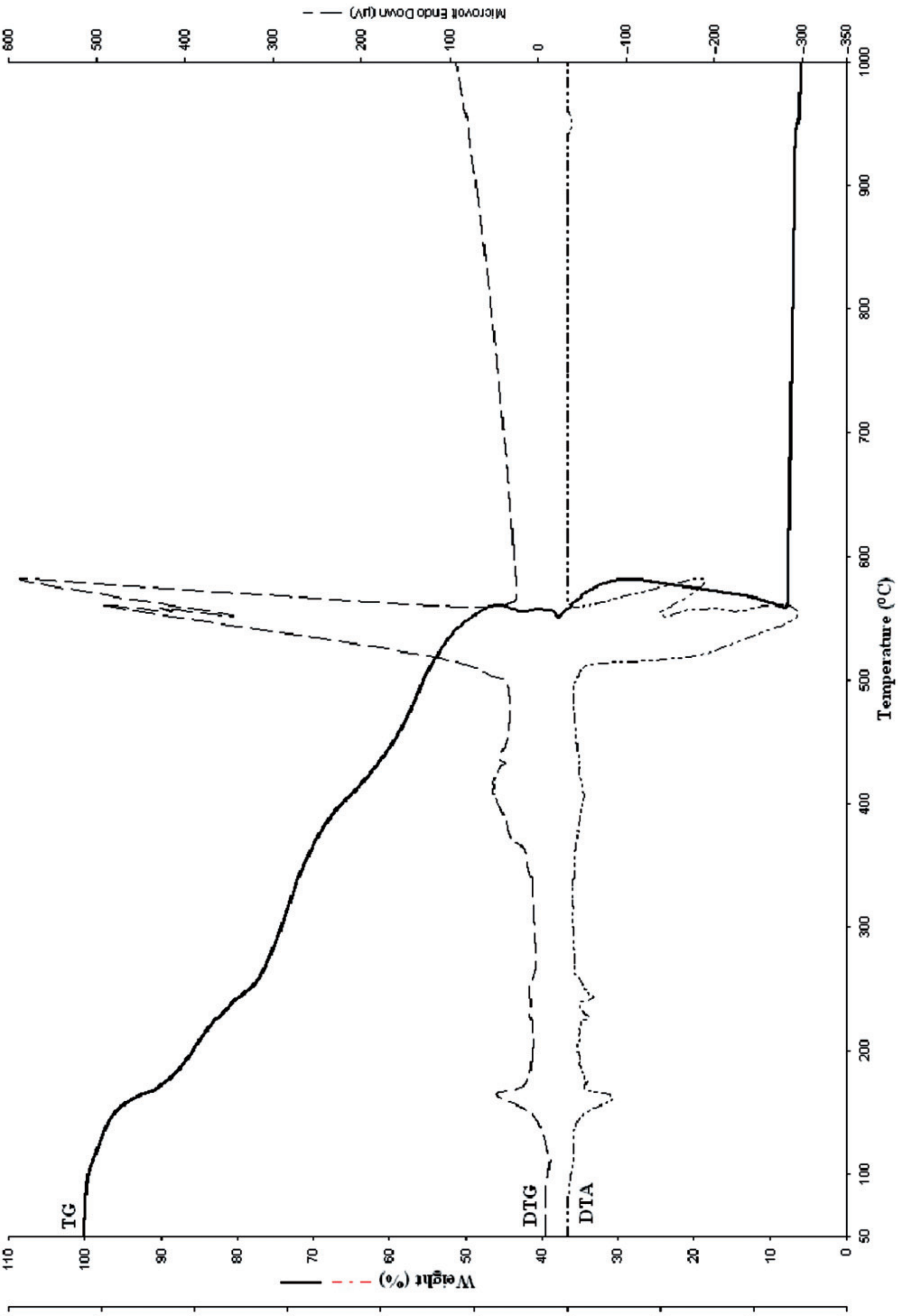


Figure 1. DTA, TG and DTG curves of **1**.

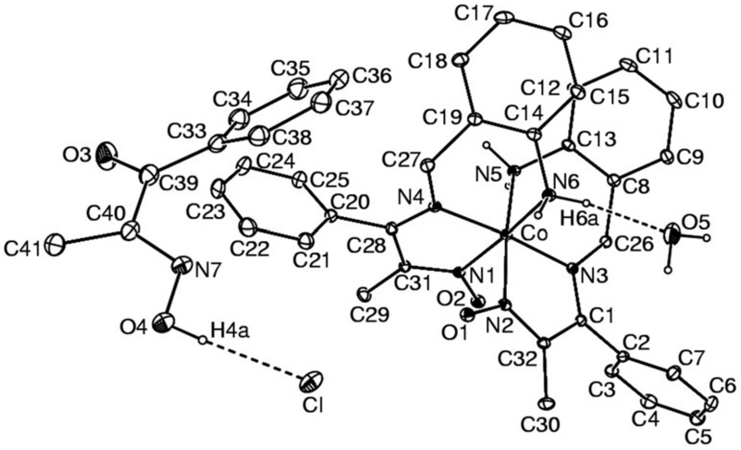


Figure 2. An ORTEP-3 [26] drawing of **1** with the atom-numbering scheme. Displacement ellipsoids are drawn at the 20% probability level. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds are omitted for clarity.

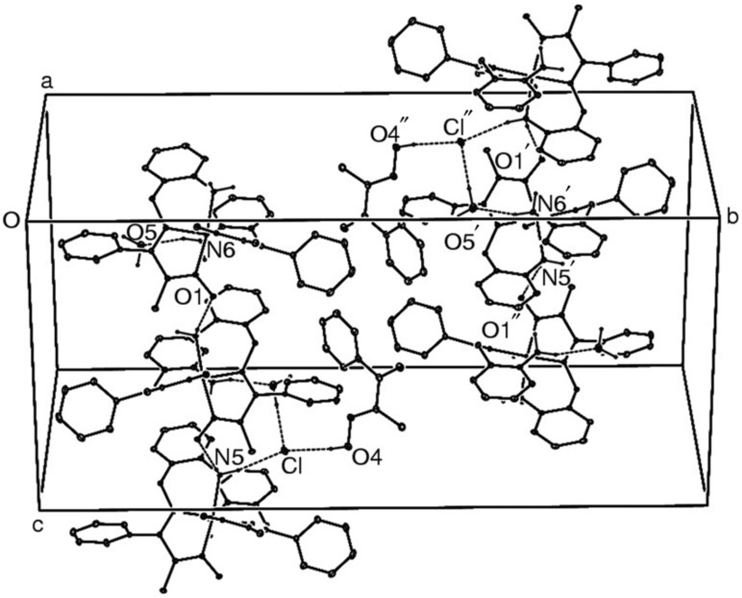


Figure 3. A partial packing diagram of **1**. Hydrogen bonds are shown as dashed lines. H atoms not involved in hydrogen bonds are omitted for clarity [symmetry codes: (′) $-x, y+1/2, -z+1/2$; (″) $-x, -y, -z$].

$\theta = 68.1(4)^\circ$ [23]. Some significant changes in the geometry of the oximes are evident when the bond lengths and angles (table 2) are compared with the corresponding values in oxime containing compounds [24]. The oxime moieties have E configurations [C1–C32–N2–O1 −175.8(3)°, C28–C31–N1–O2 −178.7(3)° and C39–C40–N7–O4 −175.3(4)°] [25]. In this configuration, the oxime groups are donors in O–H···Cl intramolecular hydrogen bonds (table 3). In the crystal structure, intramolecular O–H···Cl and N–H···O and intermolecular N–H···O, O–H···Cl and N–H···Cl hydrogen bonds (table 3) link the molecules into chains parallel to the c axis, where hydrogen bonded 1-phenyl-1,2-propanedione-2-oxime molecules fill the spaces between chains (figure 3).

## 4. Conclusion

A cobalt(III) complex, $[Co(L)_2]Cl \cdot PPO \cdot H_2O$, has been synthesized and structurally characterized. Compound **1** contains two different oximes. One is in the deprotonated form and coordinates to cobalt(III), while the other stays free in the crystal structure. Both oxime moieties have E configurations. The crystal structure exhibits intra- and inter-molecular hydrogen bonds linking the molecules into chains.

## Supplementary material

Crystallographic data (excluding structure factors) have been deposited with the Cambridge Crystallographic Data Centre as the supplementary publication No. CCDC 657960. Copies of the data can be obtained, free of charge, on application to CCDC, 12 Union Road, Cambridge CB2 1EZ, UK (Fax: +44-1223-336033 or Email: deposit@ccdc.cam.ac.uk).

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