Synthesis and Characterization of Cobalt Chelates Using 8-Hydroxyquinoline (Oxine) as a Ligand.

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ABSTRACT

Square planar complexes of cobalt in any of its common oxidation states are rare and attempt to make them either yield tetrahedral or octahedral complexes. The aim of this study was to synthesize and characterize tetrahedral or octahedral complexes of cobalt using selected chelating ligands such as 8-hydroxyquinoline (oxine) expected to have the same action as cis-platin used as anticancer drug.

The reactions of $CoCl_2$ with 8-hydroxyquinoline (oxine) were studied under different solvent conditions under inert conditions. Reaction of cobalt (II) chloride with Oxine in tetrahydrofuran (THF) solvent gave unexpected blue crystals of the tetrachloridocobaltate (II) complex $[CoCl_4]^-[C_9H_7NOH]^+$ in which protonated oxine is the counter cation.

The molecular structure of the complex $[CoCl_4]^-[C_9H_7NOH]^+$ was confirmed using single crystal X-ray crystallography. It forms monoclinic crystals in the C2/c space group with respective unit cell parameters beingα= 90.00, β= 91.0900, γ= 90.00; a = 15.1890(3), b = 7.99120(10), c = 16.6770(3).

When the same reaction was carried out in water/ethanol mixture, a yellow non crystalline solid, $[Co(oxine)_2(H_2O)_2]Cl_2$ was obtained. These compounds were characterized by melting point determination, elemental analysis and FTIR spectroscopy

Keywords: Oxine; Cis-platin; Colbalt Complexes; Tetrahydrofuran; FTIR Spectroscopy

INTRODUCTION

The discovery of the platinum-based pharmaceutical cis-platin about 4 decades ago spurred research in medicinal inorganic chemistry that is still thriving [1]. Cis-platin remains the best-selling antitumor drug in world more than 30 years since it was approved as an antitumor agent. During the last three decades, a number of coordinated metal complexes have been synthesized, characterized and tested both in cell culture and animal models for antitumor activity [2].

However, only the complexes of platinum are currently in routine clinical use. Cis-platin also known as cis-diamminedichloridoplatinum(II) (CDDP) is used to treat various types of cancers including breast, head, cervical, testicular, neck, ovarian, stomach, bladder, lung, oesophageal and prostate cancer. Complexes of platinum react in vivo, by binding and causing cross links to DNA (Figure 1.1) triggering apoptosis [1]. There are various major challenges associated with the current platinum drugs, regardless of the achievements.

These platinum drugs are active to a limited number of tumors. Intrinsic resistance is acquired by some tumors causing nausea, vomiting, suppression in bone marrow and toxicity in the kidney which are severe side-effects [3]. Although around ten other complexes of platinum are undergoing clinical trials currently, derivatives of cis-platin are still not yet able to adequately address some challenges related to cis-platin chemotherapy.

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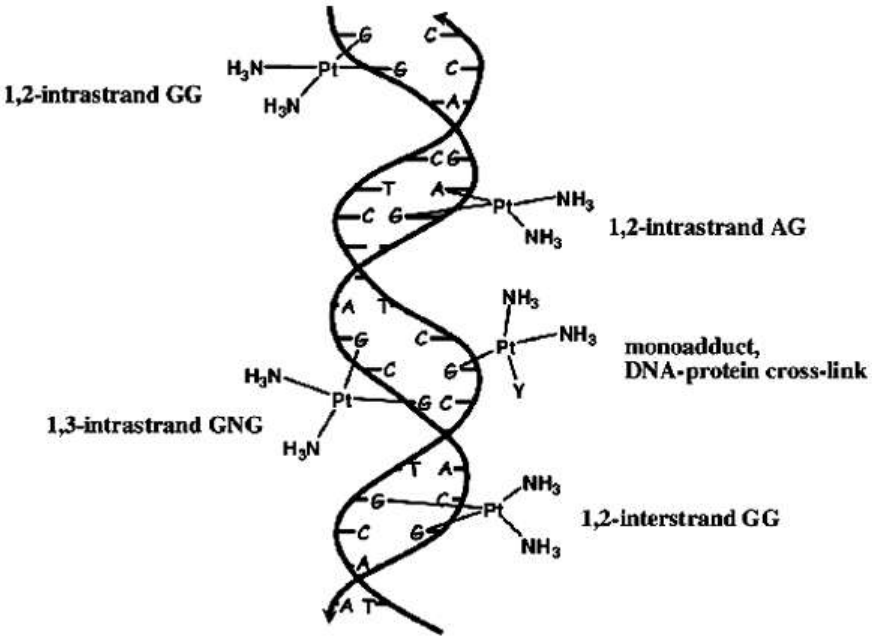

Figure 1.1: Various types of DNA-Cis-platin adducts

Cobalt forms compounds mainly in +3 and +2 oxidation states. Generally, cobalt as a metal is very rare, but it forms biologically useful coenzymes in cobalamin dependent enzymes. Cobalamin represents relatively unreactive Co (III) ion in a macrocyclic corrin ring [2]. In addition to the four nitrogen donors of the macrocyclic corrin of cobalamin coenzyme the Co (III) is coordinated to a CH_3 group or an axial 5-deoxyadenosine. Cobalamin behaves as a cofactor in a number of biochemical processes such as type II RR (which is available in archea and bacteria) and mutase methylmalonyl CoA. The number of cobalamin dependent enzymes known remains minimal. However, many organisms require vitamin B_{12} in very little amounts. The Co amounts required by human beings is about one to two grams daily, taken from our food and picked up by an elaborate absorption mechanism [2]. Selectively, vitamin B_{12} is synthesized through complex biosynthetic routes by members of the Archea and specific bacteria [4]. However, there are a few proteins which contain cobalt and not able to coordinate to the system of corrin macrocycles which have been synthesized and characterized. Uptake of cobalt in large amounts has been shown to be carcinogenic especially in rodents. Mechanisms which are underlying involve genotoxicity, radical-mediated and also as direct cobalt interference with DNA repair [5]. The production of OH from H_2O_2 in a Fenton-like reaction is catalyzed by Co (II) ion. After administering through periteneum in rats, Co (II) stimulated the production of oxidative DNA causing damage in lungs, kidney and liver [6]. Cobalt (II) ion is able to interfere with nucleotide excision repair replacing for Zn^{2+} in the proteins of zinc, such as xeroderma pigmentosum (XPA) during the process of repairing DNA [7]. The other carcinogenic effects such as benzo [α] pyrene is intensified by cobalt [8].

Compounds containing cobalt have currently attracted more research as anticancer agents despite the limitations and negative effects on tissues and normal cells. Vitamin B_{12} is administered together with folacin in chemotherapy regimens involving antimetabolites to minimize adverse effects. Fast proliferating cells need large quantities of vitamin B_{12} than normal cells. Tumor accumulation is enhanced through the respective mediated receptor- uptake system which is achieved when vitamin B_{12}-conjugates with radioisotopes or cytotoxic compounds such as nitrosyl cobalamin or cis-platin cobalamin [9].

The first structures of cobalt (III) complexes were described in 1956 [1]. Cobalt (III) complexes target specific enzymes. Activity of cobalt (III) complexes was first noted in murine leukemia cells for a complex of the acetal ligand [10]. Cobalt-alkynes analogue (Figure 1.2) of the NSAID group are extremely active against breast cancer cell lines. Compounds which were more lipophilic than their free ligands showed high intracellular levels of cobalt. It has been proposed that the potent activity was due to cyclooxygenase (COX) inhibition [11].

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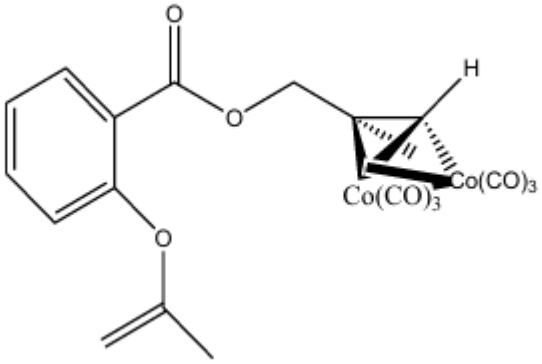

Figure 1.2: The analogue of cobalt-alkynes of

Co (III) complexes were described as selective hypoxia antitumor agents. Co (II) species may be reduced under hypoxic conditions leading to a loss of a ligand which is neutral. This idea was based on the fact that under anaerobic conditions, tumor cells develop resistance to chemotherapy agents [2]. An example of antitumor active cobalt (II) complexes is cobalt salen complex (Figure 1.3). Structure-activity relationships studies indicate that anti-proliferation depends on the position and the type of substituent in the aromatic rings [12].

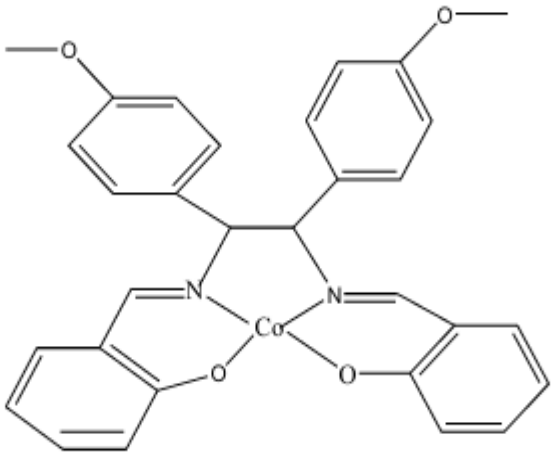

Figure 1.3: Structure of cobalt salen complex

MATERIALS AND METHODS

Materials

All preparations were done in an atmosphere of dry, oxygen-free nitrogen. Unless stated otherwise, all organic solvents were freshly distilled before use. Dichloromethane was refluxed over phosphorous pentoxide and distilled in an atmosphere of dry, oxygen-free nitrogen to further remove any traces of water. Tetrahydrofuran (THF) was refluxed over sodium metal until the benzophenone indicator turned deep purple, then distilled under inert atmosphere. Methanol and Ethanol obtained after treatment with anhydrous magnesium sulphate were refluxed over calcium oxide in an atmosphere of dry, oxygen-free nitrogen. Analytical grade 8 hydroxyquinoline (oxine) was bought from precise laboratory and allied supplies. $CoCl_2$ was bought from Kobian Kenya Limited. Single crystal XRD for the oxine complex was done at the school of Chemistry, University of Kwa-Zulu Natal, South Africa. C/H/N/O determination was done on a Perkin Elmer 2400 CHN elemental analyzer. Infrared spectra were recorded at the of range 4000 cm-1 - 400 cm-1 on an FTIR-8400 Shimadzu spectrometer. Solid samples were molded into KBr discs by grinding them (0.1-0.2%) with KBr into fine powder which was then

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pressed into transparent discs, mounted onto the spectrometer and scanned. Single crystal XRD data for one oxine complex was recorded using Bruker APEX II 4 K CCD Detectors at 100(2) K.

Gravimetric Determination of Halides

The halogens were determined gravimetrically as the silver halide [13]. The sample containing chloride ions was accurately weighed and transferred into a 250 ml beaker and digested using 5 ml of AR grade concentrated nitric acid. 100ml of the solution were transferred into a 250 ml beaker. 30 ml of 0.1M $AgNO_3$ from a burette were added to the solution drop by drop while stirring until no more precipitate formed. The beaker was covered using a watch glass then, wrapped with an aluminum foil to prevent photodecomposition of the silver halide precipitate. The precipitate was coagulated by gently warming it on a hot plate for 30 minutes. When the supernatant was clear, a few drops of 0.1mol $AgNO_3$ were added to test for completion of precipitation of the halide ions. When no turbidity was seen, the beaker was kept in a dark cupboard to cool for two hours and the filtrate separated by filtration in a pre- weighed G-4 sintered glass crucible. Residue was washed many times using very dilute HNO_3 until the filtrate gave no turbidity with 0.1M HCl. The residue on the sintered glass crucible was dried on an electric oven at 140 °C cooled in a desiccators containing phosphorous pentoxide and weighed. The process of heating, cooling and weighing was repeated until a constant weight was obtained. The determinations were carried out in duplicate.

Reaction of Cobalt (II) Chloride with Oxine in THF

A three-necked round bottomed flask equipped with a condenser was set up on a heating mantle. As dry, oxygen-free nitrogen was passing through the system, accurately weighed 5.95 g (0.025 moles) of cobalt (II) chloride was transferred into the round bottomed flask. About 20mL of THF was added. 20 ml $SOCl_2$ was poured to the solvent and the mixture refluxed for one hour to dry the solvent. 3.63 g (0.025 moles) of 8-hydroxyquinoline was weighed and dissolved in THF. The solution was added to the round bottomed flask in small portions in a period of about 10 minutes. The mixture was refluxed for 2-3hrs after which it was allowed to cool under nitrogen to room temperature. A blue precipitate was formed at the bottom of the flask. The solid was recovered by filtration and transferred to a Schlenk tube, where it was dried under reduced pressure. Melting point of the complex was determined using Gallen Kamp melting point apparatus and was found to be 217-242ºC.

Reaction of Cobalt (II) Chloride with Oxine using Water

Accurately weighed 5.93g (0.025moles) of hydrated cobalt (II) chloride was dissolved in 20mL water. 3.63g (0.025moles) of oxine was dissolved in 25 ml ethanol. The two solutions were mixed and stirred with magnetic stirrer while heating with a hot plate maintained at 65ºC for 2-4 hours. The solution was cooled until a yellow precipitate was formed which was filtered, washed using ethanol and then dried under reduced pressure. The melting point was determined in duplicate and was found to be 187-197ºC. Elemental analysis results were consistent with the formulation $CoCl_2(Oxine)_2(H_2O)_2$.

Single Crystal X-Ray Diffraction Data

Crystals of $[CoCl_4]$-$[C_9H_7NOH]^+$ suitable for X-ray diffraction studies were obtained by slow solvent evaporation. Intensity data were collected on a Bruker APEX II 4k CCD area detector diffractometer with graphite monochromatic Mo Kα radiation (λ = 0.71073 Å) (50 kV, 30 mA) using the APEX II data collection software. Data reduction was carried out using the program SAINT+ and face indexed absorption corrections were made using XPREP. "The crystal structure was solved by direct methods using SHELXTL [14]. Using SHELXTL non-hydrogen atoms were isotropically refined first followed by anisotropic refinement by full matrix least-squares calculations based on F2. First hydrogen atoms were located in the difference map then geometrically positioned and then allowed to ride on their respective parent atoms. Diagrams and publication material were generated using SHELXTL, platon and ORTEP". Table 3.3 summarizes crystal data and structure refinement information while specific bond length and angles are given in Tables 3.2.

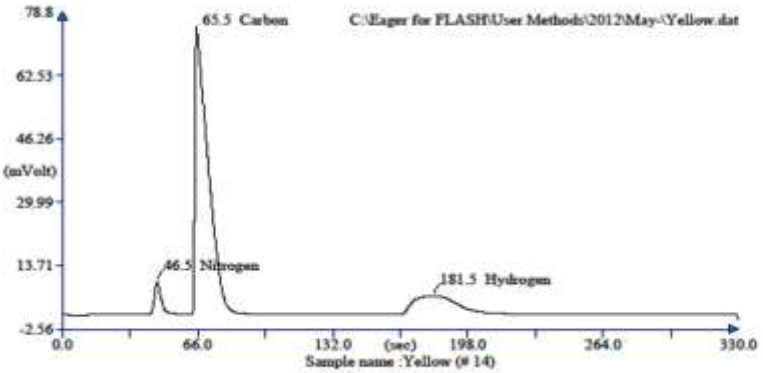
ISSN No. 2454-6194 | DOI: 10.51584/IJRIAS |Volume XI Issue V May 2026

RESULTS AND DISCUSSIONS

Elemental analysis

Table 3:1: Elemental analysis for oxine cobalt complexes

Compound	C (%)	N (%)	H (%)	O (%)
$[CoCl_4]$-$[C9H7NOH]^+$	30.04(30.4)	3.79(3.95)	2.33(2.02)	-
$CoCl_2(Oxine)2(H_2O)_2$	56.07(56.41)	7.58(7.31)	4.17(4.21)	16.35(16.70)



Retention Time (min)	Element Name	Element %
0.775	Nitrogen	7.587
1.092	Carbon	56.073
3.025	Hydrogen	4.175
		67.834

Infra-Red Absorption Data.

Reaction of Cobalt (II) Chloride with Oxine in THF using $SoCl_2$

Reaction of $CoCl_2$ with oxine in 1:1 mole ratio in THF at 87ºC, resulted in the formation of a blue solid which was found to be $[CoCl_4]$-$[C_9H_7NOH]^+$ in which the oxine ligand was not coordinated to the metal centre. The excess Cl^- might have come from the hydrolysis of the $SOCl_2$ to give HCl and SO_2. Acid protonates the N-H making it unable to coordinate to the metal centre. The molecular structure was confirmed by single crystal x-ray diffraction. The solid was soluble in THF and was recrystallized from THF at room temperature. The solid melted without decomposition at 217-242ºC. The molecular structure of the compound was confirmed by single crystal X-ray crystallography. The crystal was bimolecular and crystallized in monoclinic crystal 28 system. It was refined to an r value of 0.0226. IR spectrum of the complex showed strong bands assignable to aromatic C=N, C=C and C-O stretching modes at 1662cm^{-1}, 1495 cm^{-1}, and 1268 cm^{-1} respectively. Compared to free oxine ligand strong band of C=N, C=C and C-O were found at 1602 cm^{-1}, 1496 cm^{-1} and 1275 cm^{-1} respectively. There was no change in the position of the bands in Oxine complex as compared to that of the ligand. The band at 1269 cm^{-1} shows presence of O-C group. Bands at 405 cm^{-1} and 504 cm^{-1} due to M-N and M-O stretching vibrations were missing from the IR spectrum of the complex meaning the ligand was not coordinated to the metal ion.

Reaction of Cobalt (II) Chloride with Oxine in Water

Reaction of $CoCl_2$ with oxine in 1:1 mole ratio in water at 65ºC, resulted in the formation of a yellow solid which was found to be $[Co(oxine)_2(H_2O)_2]Cl_2$ from elemental and gravimetric analysis. This solid is soluble in water

ISSN No. 2454-6194 | DOI: 10.51584/IJRIAS |Volume XI Issue V May 2026

at room temperature. The solid melted without decomposition at 187-197ºC The IR spectrum of the complex shows new bands at 450 cm^{-1} corresponds to ν (M-O) and 503 cm^{-1} corresponds to ν (M-N), which are not found in the spectrum of the free oxine. The carbon-oxygen (C-O) vibration bands at 1104.25 cm^{-1} show a frequency change due to change of electronic distribution in the environment of the C-O band on chelating. The vibration bands in the region of 1650 cm^{-1} to 1400 cm^{-1} of C=N and C=C stretching vibration are shifted to the lower frequencies in the spectra of the metal complexes than the respective bands in the oxine. The shift could be explained on the assumption that during chelating, the electron distribution of the quinoline ring is disturbed slightly and partially due to chelation. The usual ring skeleton ν(C-C) and ν(C-N) bands are observed around 1600 cm^{-1}, 1450 cm^{-1}, and 1370 cm^{-1}. In the chelates the band observed around 450 cm^{-1} corresponds to ν 29 (M-O), suggesting that phenolic groups are involved in bond formation with metal ion and 503 cm^{-1} corresponds to ν (M-N). One more band in the range of 750-830 cm^{-1} attributable to rocking and wagging modes of vibration of water (H_2O) was present in the coordination complex but not in the free oxine ligand [15].

Structural Characterization of $[CoCl_4]$-$[C_9H_7NOH]^+$

Crystals of $[CoCl_4]$-$[C_9H_7NOH]^+$ were obtained as blue-green blocks that crystallized in the monoclinic C2/c space group, with one anion and two cations in the asymmetric unit (see packing in Figure (3.1).

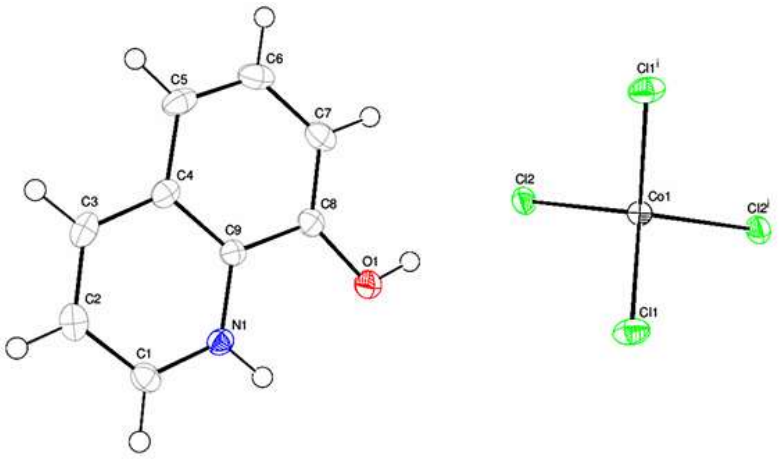


Figure 3.1: ORTEP diagram of [CoCl4]-[C9H7NOH]+

The bond angles around the Co metal Cl(1) Co(1) Cl(2) (112.65) shows slight deviations from the tetrahedral angles of 109° giving the crystal a distorted tetrahedral geometry. Figure 3.2 illustrates the parking of the molecules in the crystal viewed down the axis b. The molecules are 30 arranged in layers and the asymmetric units alternate in orientation along the c axis such that the pairs of cations have the OH groups facing opposite directions in a staggered manner.

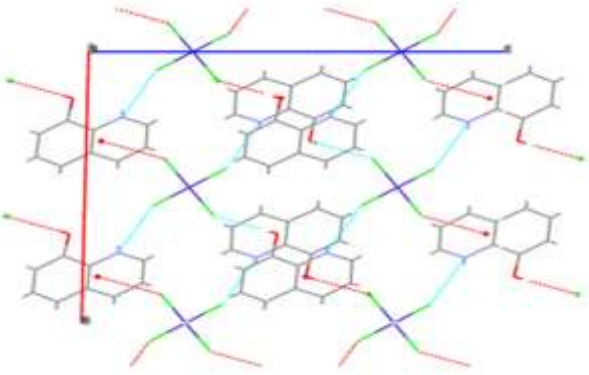


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Figure 3.2: The parking diagram for $[CoCl_4]$-$[C_9H_7NOH]^+$ viewed along b axis

Figure 3.3 shows the hydrogen bonds between the cations and anions in the structure. Each anion is surrounded by four cation pairs which are in staggered orientation such that the anion is hydrogen-bonded to four different cations from the four pairs i.e. two Cl atoms are hydrogen bonded to the OH groups while the remaining two are hydrogen-bonded to the NH groups of the cations. The remaining four cations are hydrogen bonded to four of the surrounding $[CoCl_4]$- chlorides.

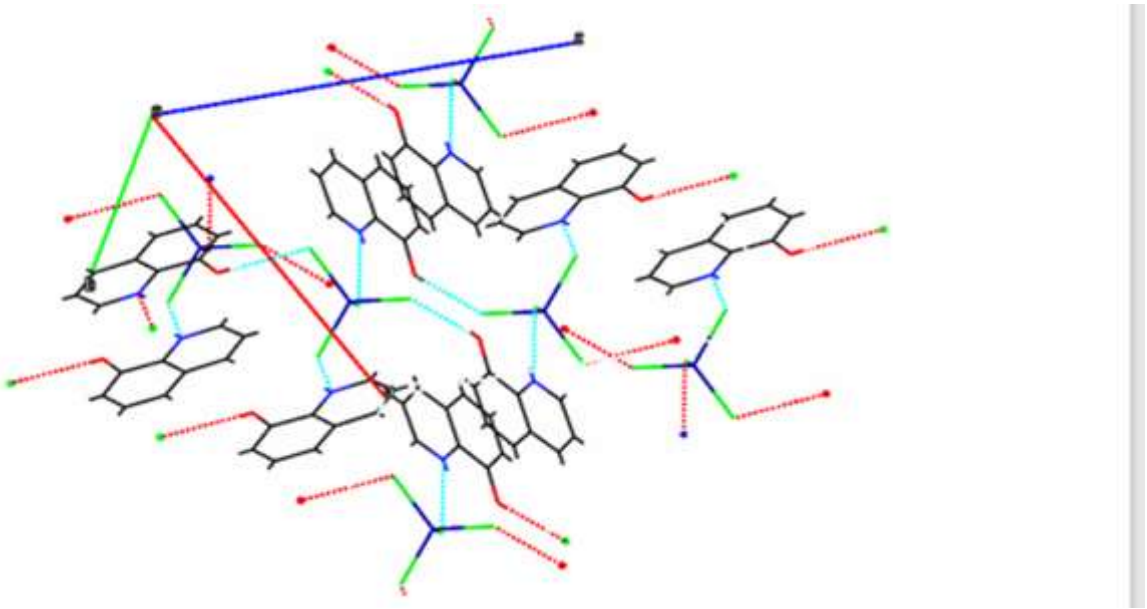

Figure 3.3: The parking diagram for $[CoCl_4]$-$[C_9H_7NOH]^+$ showing H-bonds

The bond distances and angles are given in table 2 and the parameters for crystal data in Table 3.2. The Co1-Cl1 bond lengths are notably longer than Co1-Cl2. The average bond between the metal Co and the surrounding chlorides Cl1 and Cl2 does not compare well with those reported for a similar complex [12].

Table 3:2: Bond lengths (Å) and angles (°) for $[CoCl_4]$-

Bond	Length(Å)	Bond	Angles(°)
C1-N1	1.3263(18)	N1-C1-C2	120.30(13)
CI-C2	1.395(2)	N1-C1-H1	119.9
C1-H1	0.9500	C2-C1-H1	119.9
C2-C3	1.369(2)	C3-C2-H2	120.4
C2-H2	0.9500	C1-C2-H2	120.4
C3-C4	1.413(2)	C2-C3-C4	121.03
C3-H3	0.9500	C2-C3-H3	119.5
C4-C9	1.4111(18)	C4-C3-H3	119.5
C4-C5	1.4170(19)	C9-C4-C3	117.67(13)
C5-C6	1.371(2)	C9-C4-C5	118.51(13)
C5-H5	0.9500	C3-C4-C5	123.82(13)
C6-C7	1.410(2)	C6-C5-C4	119.41
C6-H6	0.9500	C6-C5-H5	120.3
C7-C8	1.3730(19)	C4-C5-H5	120.3
C7H7	0.9500	C5-C6-C7	121.58(13)
C8-O1	1.3579(17)	C5-C6-H6	119.2
C8-C9	1.4145(18)	C7-C6-H6	119.2
Cl1-Co1	2.2731(3)	Cl1-Co1-Cl1	104.64(12)
Cl2-Co1	2.2835(3)	Cl1-Co1-Cl2	107.59(12)
Co1-Cl1	2.2731(3)	Cl1-Co1-Cl2	112.65(12)
Co1-Cl2	2.2835	Cl1-Co1-Cl2	112.65(12)
O1-H1	0.8400	C8-O1-H1	109.5

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Table 3.3: Crystallographic data for [CoCl4]-[C9H7NOH] +

Empirical formula	$C_9H_{16}Cl_4CoNO$
Chemical formula moiety	$2(C_9H_8NO)Cl_4Co$
Formula weight	493.06
Temperature	100(2) K
Crystal system	Monoclinic
Space group	C2/c
Unit cell dimensions	a = 15.1890(3),α =90.00, b =7.99120(10) β =91.0900, c =16.6770(3), γ =90.00
Volume	2023.59(6) cm^3
2 Calculated density	(4) g/cm^3
Absorption coefficient	1.392 cm^{-1}
F(100)	996
Crystal size	max (0.17)min(0.04)
Theta range for data collection	2.44-28.28
Goodness-of-fit on F^{-2}	1.096
R indices (all data)	0.022
Extinction coefficient	None

CONCLUSION

The Co (II) chloride salt reacts with chelating ligands to give high yields of the chelates but the kind of complex obtained depends on the conditions of synthesis. When hydrated cobalt (II) chloride reacts with the chelating ligand Oxine in THF in the presence of thionyl chloride, the result was the unexpected Co (II) complex $[CoCl_4]$-$[C_9H_7NOH]^+$ in which protonated oxine acts as the counter anion and not a ligand. Reaction of cobalt (II) salt with the same ligand in water/ethanol solvent mixture gave $[Co(oxine)_2(H_2O)_2]Cl_2$ in which the metal retained oxidation state (II) and the chelating ligand is coordinated to the metal.

Conflict of interest

The authors declare that they have no conflict of interest

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