

## Research Article

# Synthesis, Characterization, and Crystal Structure of $[\text{Co}_4(\text{CH}_3\text{CO}_2)_2\text{L}_4]_2[\text{BPh}_4]_4 \cdot 0.5\text{H}_2\text{O}$ , Where HL = 4-(Salicylaldiminato)antipyrine

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The title complex was isolated as a red solid from the reaction of 4-(salicylaldiminato)antipyrine, HL, and cobalt (II) acetate in ethanol. The complex has been characterized by elemental analysis, FTIR, UV-Vis, and X-ray single crystal diffraction. Two crystallographically different cationic units, A and B, of the title complex are found. Both units are essentially isostructural; nevertheless, small differences exist between them. Both units contain four cobalt atoms arranged at the corners of distorted cubane-like core alternatively with phenoxy oxygen of the Schiff base. In both cases, one cobalt binds to three coordinated sites from the corresponding tridentate Schiff base ligand, and the fourth one was bonded by the acetate oxygen, and the fifth and the sixth donor sites come from the phenolate oxygen of another Schiff base ligand.

## 1. Introduction

Polynuclear as well as binuclear Co(II) complexes have attracted much attention due to their potential advantages than mononuclear complexes toward the preparation of molecular magnets and their application in data storage and memory devices [1]. The discovery of single molecule magnetism (SMM) in high-spin Ni(II) molecular clusters, particularly in cubane-like tetranuclear Ni(II) complexes, revived the interest in such compounds in order to study the correlation between the magnetic anisotropy of the high-spin ground state and the magnetization at low temperatures [2]. In comparison to mononuclear complexes, binuclear and polynuclear complexes could also provide more than one metal active center as Lewis acid in catalytic process, which is the interest of many researchers [3].

Crystal structures of complexes containing  $\text{Co}_4\text{O}_4$  cubane-like core, as  $[\text{Co}_4\text{O}_4(\text{OAc})_2(\text{bpy})_4](\text{ClO}_4)_2$ ,

$[\text{Co}_4(\text{CH}_3\text{O})_4(\text{C}_5\text{H}_7\text{O}_2)_4(\text{C}_2\text{H}_3\text{O}_2)]$ , and  $[\text{Co}_4\text{O}_4(\text{C}_8\text{H}_9\text{N}_2\text{O}_2)] \cdot 7.5\text{H}_2\text{O}$  [4], are well-known for several decades. Polynuclear metal complexes with tridentate ligands containing at least one hydroxyl group and oxygen as terminal coordinating atom have been reported and attracted much attention [5]. These ligands often form polynuclear complexes with cubane or double cubane structure with missing one vertex each [6]. Antipyrine and its derivatives are one of such compounds that act as tridentate ligands, and this type of ligands has been attractive to researchers, since they are used as antifever and pain relieving drugs [7].

Transition metal Schiff base complexes have been also used as antifungal and antibacterial reagents [8]. It is shown that transition metal complexes containing 4-aminoantipyrine as a Schiff base have anticancer and antibacterial activity [9] and have an important effect on simulate enzymes [10]. The type of the metal salt and the ligand plays an important role in tailoring the final product. Here,

we report the first type of the Co(II) complex that contains two crystallographically different ions 1.a and 1.b, with tetranuclear cobalt atoms each. The Co(II) atoms reside in the corners of a cubane-like structure alternatively with oxygen atoms from the Schiff base ligands.

## 2. Experimental

**2.1. Material and Measurement.** All reagent grade chemicals used in this work were obtained commercially from Aldrich or BDH and used without any further purification. All manipulations were carried out under atmospheric pressure. Elemental analysis (C, H, and N) was performed on a Vario El(III) elemental analyzer. FTIR spectra were recorded at room temperature with a Bruker IFS-25 OPUS/IR over the range from 400 to 4000  $\text{cm}^{-1}$  with resolution of 4  $\text{cm}^{-1}$ . The electronic absorption spectrum was recorded over the range 200–800 nm using Cary 5000 UV-VISNR spectrophotometer.

**2.1.1. Synthesis of (4-Salicylaldiminato)antipyrine-EtOH (HL-EtOH).** Yellow crystals of Schiff base, HL, were prepared using previously described method [11, 12]. A mixture of 4-aminoantipyrine (2.0 g, 9.85 mmol) in 20 mL ethanol and salicylaldehyde (1.22 g, 10 mmol) in 10 mL ethanol were refluxed together. Complete reaction with near quantitative conversion to the product required a period of 2.0 h. After cooling to room temperature, the yellow precipitate formed was collected by suction filtration and recrystallized from hot ethanol as deep yellow microcrystals (yield 2.81 g, 85%). The Schiff base was characterized by elemental analysis (Anal. Calc. for  $\text{C}_{20}\text{H}_{22}\text{N}_3\text{O}_3$ : C, 68.18; H, 6.25; N, 11.93; Found: C, 67.95; H, 6.25; N, 11.70).

**2.1.2. Synthesis of  $[\text{Co}_4(\text{CH}_3\text{CO}_2)_2\text{L}_4]_2[\text{BPh}_4]_4 \cdot 0.5\text{H}_2\text{O}$ .** A solution of HL (2.0 mmol, 0.6 g) in 15 ml ethanol and cobalt (II) acetate (2.0 mmol) in 15 mL ethanol were refluxed together; after 2 h, a red solid material was precipitated. The resulting solid was separated by filtration, washed with ethanol and dichloromethane, and air-dried. The product was dissolved in hot methanol and a suitable methanol solution of sodium tetraphenylborate was added and stirred for a while to give the title complex as a red solid. The final product was dissolved in acetone and filtered; on slow evaporation, red crystals were formed (yield 62%). The final product was characterized by elemental analysis, FTIR, UV-Vis, and single-crystal X-ray diffraction analysis (Anal. Calc. for  $\text{C}_{248}\text{H}_{219}\text{B}_4\text{Co}_8\text{N}_{24}\text{O}_{24.5}$ : C, 66.99; H, 4.93; N, 7.56. Found: C, 67.38; H, 5.4; N, 7.23).

**2.1.3. Crystal Structure Determination and Refinement of the Title Complex.** Suitable red single crystals of the complex were obtained by slow evaporation of an acetone solution at room temperature. X-ray single-crystal diffraction data were collected on an Oxford Gemini S diffractometer equipped with CCD detector at 295 K; MoK $\alpha$  radiation ( $\lambda = 0.71073 \text{ \AA}$ ) was used and a multiscan correction for absorption was applied. The structure was solved by direct methods (SIR92) [13] and refined on  $F^2$  by full matrix least-squares SHELXL97

[14] and WinGX [15]. All nonhydrogen atoms except O<sub>25</sub> were only partially occupied (s.o.f = 0.5) and their H atoms were not located. Positions of hydrogen atoms bonded to C were calculated geometrically and refined by a riding model. All aromatic C<sub>6</sub>-rings are constrained to be planar using FLAT instruction. Although there were indications that some rings are disordered, this behavior was not further explored due to very large number of atoms in the asymmetric unit. Data collection: CrysAlis PRO [16], cell refinement: CrysAlis PRO; data reduction: CrysAlis PRO; program(s) used to solve structure: SIR97 [17], program(s) used to refine structure: SHELXL97 [14], molecular graphics: ORTEP-3 for Windows [18], and Mercury [19], software used to prepare material for publication: publCIF [20], and PARST [21]. Crystal Data. The details of the crystallographic data and structure refinement for the complex are shown in Table 1. Additional material available from the Cambridge Crystallographic Data Center comprises thermal parameters and remaining bond distances and angles (CCDC number 801603).

**2.2. Crystal Data.** See Table 1.

## 3. Results and Discussion

A novel cobalt complex  $[\text{Co}_4(\text{CH}_3\text{CO}_2)_2\text{L}_4]_2[\text{BPh}_4]_4 \cdot 0.5\text{H}_2\text{O}$  was synthesized by the reaction of cobalt (II) acetate with HL Schiff base in ethanolic solution. The IR spectrum of the complex exhibited strong absorptions at 1625 and 1562  $\text{cm}^{-1}$  assignable to the carbonyl group of the pyrazolone ring,  $\nu(\text{C}-\text{O})$ , and azomethine group ( $\text{HC}=\text{N}$ ) of "L," respectively. The first absorption band of the complex was shifted to the lower frequency for about 29  $\text{cm}^{-1}$  relative to the free ligand (1654  $\text{cm}^{-1}$ ) indicating that the ligand coordinates through the carbonyl oxygen of the pyrazolone ring. The second absorption was shifted to the lower frequency for about 29  $\text{cm}^{-1}$  (free ligand at 1591  $\text{cm}^{-1}$ ) suggesting the involvement of the nitrogen atom of the azomethine group in the coordination. The absorptions appears as shoulders at 1610 and 1457  $\text{cm}^{-1}$  indicates the asymmetric and symmetric stretching frequencies of the acetate  $-\text{COO}^-$  groups. The separation (143  $\text{cm}^{-1}$ ) between the two absorptions indicates that the acetate group acts as a bidentate bridging ligands [22]. The  $\nu(\text{OH})$  absorption at 3446  $\text{cm}^{-1}$  indicates the presence of water molecule. The tetraphenylborate showed a finger print absorption at 2927–3044  $\text{cm}^{-1}$ . The UV-Vis spectrum of the title complex was obtained in acetone solution. The spectrum shows a very intense band at 202  $\text{cm}^{-1}$  due to  $\pi-\pi^*$  transition characteristic for aromatic rings, and another intense band at 232 nm corresponding to the charge transfer band. The bands at 324 nm with a shoulder at 315 nm and 401 nm are assigned to the d-d transitions of the high-spin Co(II) ions in an octahedral geometry. There is a blue shift in this bands compared with the absorption spectra of some hexacoordinate Co(II) complexes with the peak maxima wavelength ( $\lambda_{\text{max}}$ ) at about 500–513 nm [23].

The asymmetric units of the title complex contain two tetranuclear cations, A and B, of the formula  $[\text{Co}_4(\text{CH}_3\text{CO}_2)_2\text{L}_4]^{2+}$  with a cubane-like  $\text{Co}_4\text{O}_4$  core. In addition, four  $[\text{BPh}_4]^-$  ions and one water molecule

TABLE I: Crystal data and structure refinement details for the complex.

Empirical formula	$C_{248}H_{219}B_4Co_8N_{24}O_{24.50}$
Formula weight	4442.15
Temperature	295 K
Wavelength	0.71073 Å
Crystal system	Triclinic
Space group (number)	P-1 (2)
Unit cell parameters	$a = 19.4643(5)$ Å, $\alpha = 98.095(2)^\circ$ $b = 22.3372(5)$ Å, $\beta = 107.063(2)^\circ$ $c = 29.1735(5)$ Å, $\gamma = 109.257(2)^\circ$
Cell volume	11043.4(4) Å <sup>3</sup>
Z	2
$\rho_c$	1.336 g cm <sup>-3</sup>
$\mu$	0.659 mm <sup>-1</sup>
$F(000)$	4614
Crystal size	0.48 × 0.25 × 0.18 mm <sup>3</sup>
$\theta$ range for data collection	2.88–25.68°
Index ranges	$-23 \leq h \leq 23$ , $-27 \leq k \leq 26$ , $-32 \leq l \leq 35$
Reflections collected	98826
Independent reflections	41906 [ $R(\text{int}) = 0.0725$ ]
Completeness	99.8%
Data/restraints/parameters	41906/96/2772
Goodness-of-fit on $F^2$	1.023
R indices	$[I > 2\sigma(I)]R_1 = 0.0816$ , $wR_2 = 0.1484$
R indices (all data)	$R_1 = 0.1632$ , $wR_2 = 0.1826$
Largest difference peak and hole	0.681 and $-0.350$ eÅ <sup>-3</sup>

with 0.5 occupancy are present. The cations are close to be isostructural, and they only slightly differ in corresponding bond distances and angles. Crystal structure of the title complex with atomic numbering scheme are presented as ORTEP (Figure 1), the stereoview of the cell contents of the title complex was shown in (Figure 2) and relevant bond distances were given in Table 2. From ORTEP presentation and molecular packing diagram (Figure 3), the ligand, “L,” acts as trichelate coordinating to the Co atom by two O and one N atoms. The oxygen of the phenolate group further bridges three Co(II) ions ( $\mu_3$ -O) in the cubane-like core. Both cationic units contain Co(II) residing in a pseudooctahedral environment with  $CoNO_5$  chromophore. Each complex cation can be regarded as a dimer of dimeric units composed by phenoxy bridged dicobalt (II) subunits. The basal-planes of Co(II) include two oxygen atoms from the phenolate groups of two “L” ligands and another oxygen atom from the pyrazolone ring; the fourth position was occupied by the azomethine nitrogen of “L.” Axial sites are occupied by two oxygen atoms, one from the acetate group and the other one from another phenolate group of “L.”

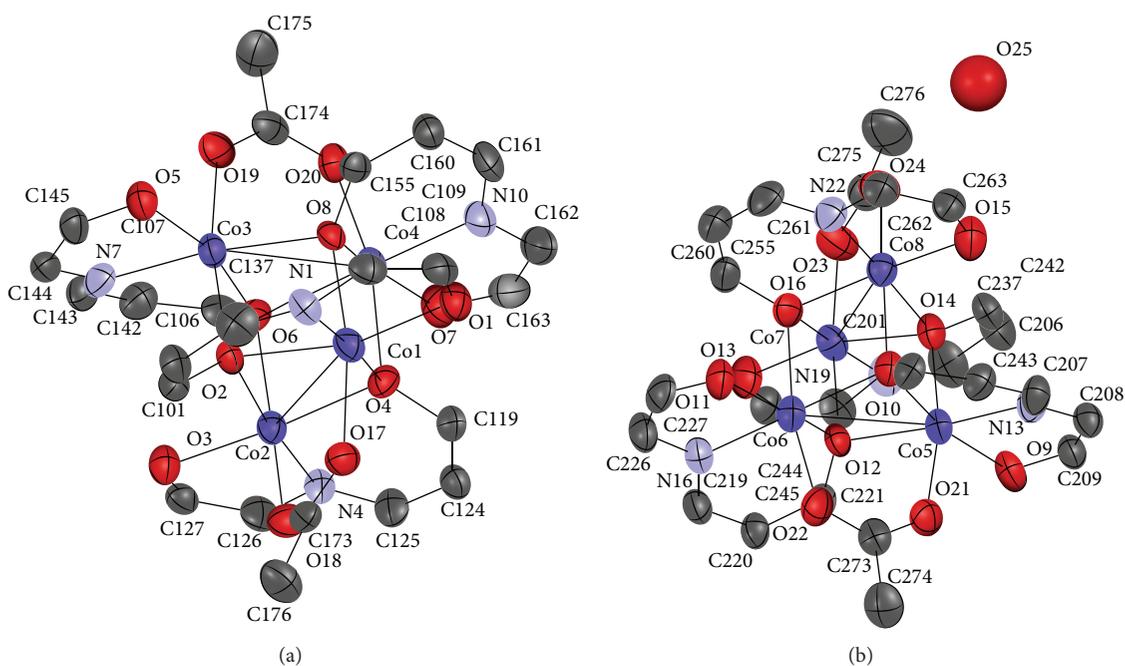
The acetate group acts as triatomic bidentate ligands bridging two Co atoms in their familiar syn-syn manner on opposite faces of the cube and the ligand “L” chelates the remaining four faces. The two faces bridged by acetate group in cationic unit A exhibit shorter  $Co \equiv Co$  separations of 2.996 and 3.022 Å, and more acute  $Co-O-Co$  angles ranging from 89.06 to 90.03° with  $Co-O-O-Co$  dihedral angles of

154.16 and 156.56° compared with those not bridged by acetate group, where  $Co \equiv Co$  separations are in the range of 3.24 to 3.31 Å,  $Co \equiv Co$  angles in the range of 97.26 to 102.28°, and  $Co-O-O-Co$  dihedral angles in the range of 177.06 to 178.99°. Cationic unit B is a little bit shorter in  $Co \equiv Co$  separations with more acute angles. These variations in bond distances and angles between faces of the cubane-like cores are responsible for their distortions. This distortion produces two types of  $Co-O-Co$  angles, those that are  $<90^\circ$  at the top and bottom faces of the core and those that are  $>101^\circ$  at the rest of the cubane faces. Therefore, one can expect that the Co(II) ions with small angles will lead to some magnetic properties comparable to the  $[Ni_4(OCH_3)_4(dbm)_4(MeOH)_4]$ , dbm = dibenzoylmethane [24].

The average  $Co-OAc$  bond distances of the title complex of 2.02 Å are longer than that reported for  $[Co_4O_4(dpah)_4(CH_3CO_2)_2]_2V_4O_{12} \cdot 5H_2O$  (1.98 Å) and shorter than that reported for  $[Ni_4(OCH_3)_4(OAc)_2(TMB)_4](BPh_4)_2 \cdot 4CH_2Cl_2$ , TMP = 2,5-dimethyl-2,5-diisocyanohexane (2.05 Å) [25]. The bond distances of  $Co-OAc$  in cationic unit B are a little bit longer than in cationic unit A by 0.02 Å. The difference in all bond distances of the acetate groups in both units ranges from 0.01 to 0.02 Å and is very close to that reported for Ni(II) complex [25]. While the phenolate oxygen atom is ligating three cobalt atoms in a tetrahedral environment, the four cobalt atoms are located at four alternative corners of a distorted cubane-like core and the rest of the cubane vertices are occupied by four

TABLE 2: Selected bond lengths (Å) and angles (°) of the title compound.

Bond lengths					
Co1–O17	2.045(5)	Co1–O2	2.107(4)	Co1–O1	2.107(4)
Co1–N1	2.109(6)	Co1–O8	2.154(4)	Co1–O4	2.157(4)
Co2–O18	2.030(5)	Co2–O4	2.087(4)	Co2–N4	2.091(6)
Co2–O3	2.110(4)	Co2–O6	2.150(5)	Co2–O2	2.201(4)
Co3–O19	2.014(5)	Co3–N7	2.083(5)	Co3–O6	2.108(4)
Co3–O2	2.145(4)	Co3–O5	2.152(5)	Co3–O8	2.152(4)
Co4–O20	2.015(5)	Co4–O8	2.084(4)	Co4–N10	2.098(6)
Co4–O7	2.122(5)	Co4–O6	2.154(4)	Co4–O4	2.208(5)
Co5–O21	2.005(5)	Co5–N13	2.099(6)	Co5–O10	2.113(4)
Co5–O14	2.136(5)	Co5–O9	2.146(5)	Co5–O12	2.176(4)
Co5–Co6	2.9972(13)	Co6–O22	2.029(5)	Co6–O12	2.091(5)
Co6–N16	2.094(6)	Co6–O11	2.126(5)	Co6–O10	2.144(5)
Co6–O16	2.231(5)	Co7–O23	2.041(6)	Co7–O13	2.098(5)
Co7–O14	2.098(4)	Co7–N19	2.110(6)	Co7–O12	2.155(5)
Co7–O16	2.159(5)	Co7–Co8	2.977(14)	Co8–O24	2.035(6)
Co8–O16	2.067(5)	Co8–O15	2.074(5)	Co8–N22	2.100(6)
Co8–O14	2.176(5)	Co8–O10	2.181(5)		
Bond angles					
Co1–O2–Co3	101.32(17)	C98–O2–Co2	122.8(4)	Co1–O2–Co2	89.06(15)
Co3–O2–Co2	96.65(17)	Co2–O4–Co1	90.78(16)	Co2–O4–Co4	99.85(17)
Co1–O4–Co4	97.17(16)	Co3–O6–Co2	99.34(19)	Co3–O6–Co4	89.33(16)
Co2–O6–Co4	99.59(17)	Co4–O8–Co3	90.03(16)	Co4–O8–Co1	101.14(17)
Co3–O8–Co1	99.57(16)	Co5–O10–Co6	89.51(17)	Co5–O10–Co8	97.97(19)
Co6–O10–Co8	100.00(18)	Co6–O12–Co7	102.1(2)	Co6–O12–Co5	89.23(17)
Co7–O12–Co5	98.95(18)	Co7–O14–Co5	102.1(2)	Co7–O14–Co8	88.28(17)
Co5–O14–Co8	97.4(2)	Co8–O16–Co7	89.53(19)	Co8–O16–Co6	100.78(19)
Co7–O16–Co6	97.59(19)				

FIGURE 1: An ORTEP drawing of  $[\text{Co}_4\text{L}_4(\text{OAc})_2]^{2+}$  cationic units (a) and (b) of the title complex with 50% probability thermal ellipsoids showing the atom labeling scheme. Hydrogen atoms are omitted for clarity.

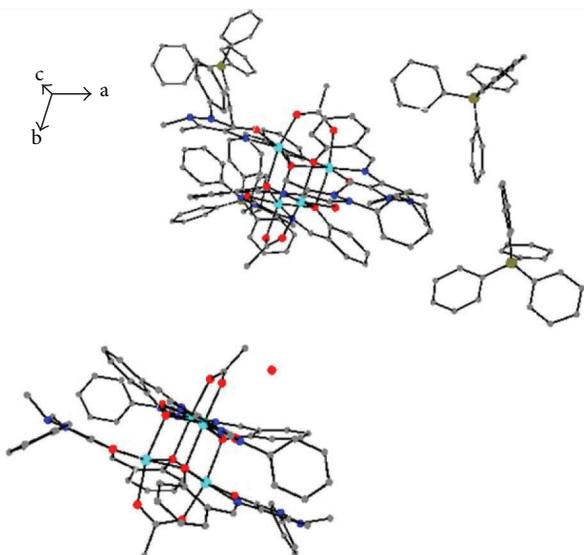


FIGURE 2: Stereoview of the unit cell contents of  $[\text{Co}_4\text{L}_4(\text{OAc})_2]_2(\text{BPh}_4)_4 \cdot 0.5\text{H}_2\text{O}$ . Hydrogen atoms are omitted for clarity.

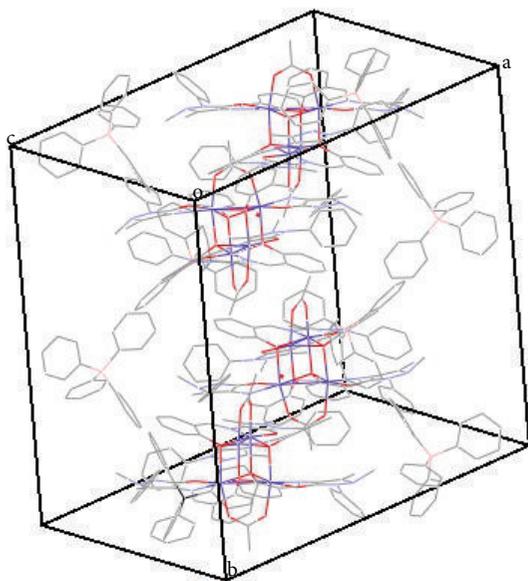


FIGURE 3: View of the molecular packing diagram showing 3D structure of the title complex. Hydrogen atoms are omitted for clarity.

oxygen atoms from four L as  $\mu_3$ -O. Both units (Figure 1), as we mentioned before, differ slightly in corresponding bond distances and angles; even in the cubane itself the separations between cobalt atoms differ slightly, for example, the longest separation for cationic unit A of  $\text{Co}_2\equiv\text{Co}_4$  (3.288 Å) and the shortest separation of  $\text{Co}_3\equiv\text{Co}_4$  (2.996 Å), for cationic unit B, the longest separation of  $\text{Co}_6\equiv\text{Co}_8$  (3.313 Å), and the shortest of  $\text{Co}_7\equiv\text{Co}_8$  (2.979 Å). The shortest  $\text{Co}\equiv\text{Co}$  separation in the title complex is still longer than the longest one reported for  $[\text{Co}_4\text{O}_4(\text{dpah})_4(\text{OAc})_2]_2\text{V}_4\text{O}_{12} \cdot 5\text{H}_2\text{O}$ , dpah = 2,2-dipyridylamine (2.848 Å) [26]. The average

$\text{Co}\equiv\text{Co}$  separations in both units are very close to the separation in  $[\text{Co}_2\text{L}_2(\text{NCS})_2(\text{CH}_3\text{OH})_2]$  (3.1028 Å) [27] and in  $[\text{Co}_2\text{L}_2\text{Cl}_2(\text{CH}_3\text{OH})_2]$  (3.085 Å) [3], where “L” is the same Schiff base in the first and 2-hydroxyisophthaldehyde oxime in the second case. The C–O bond distances in the phenolate groups of the ligands, which act as  $\mu_3$ -O bridges, range from 1.377(5) to 1.381(5) Å in cationic unit A and from 1.367(5) to 1.379(5) Å in cationic unit B. All of these distances are still longer than C–O distance of the phenolate group which acts as  $\mu_2$ -O in  $[\text{Co}_2\text{L}_2(\text{NCS})_2(\text{CH}_3\text{OH})_2]$  (1.330(2) Å) and even longer than the distance of the free Schiff base (1.345 Å) [11, 12].

Further analysis of the crystal structure revealed that this structure contains a half water molecule in the lattice. There is a short contact between  $\text{O}_{25}$  and  $\text{H}_{27}\text{E}$  atom belonging to the acetate methyl group at 3.01(1)8 Å and another  $\text{O}_{24} \cdots \text{O}_{25}$  contact of 2.90(2) Å indicating possible hydrogen bonds. The four counter ions  $[\text{BPh}_4]^-$  exhibit their normal geometry. The two A and B cationic units of the title complex are held together by an electrostatic attraction with  $[\text{BPh}_4]^-$  ions. Finally, similar complexes of Ni(II), Mn(II), mixed Co(II) and Ni(II), and mixed Co(II) and Cu(II) complexes were prepared and characterized by spectroscopic methods and their structures will be investigated in near future.

#### 4. Conclusion

From all of these, we conclude that the novel Co(II) complex has two very large A and B cationic unit complexes with a cubane-like core; A and B units are almost isostructural and differ slightly in bond distance and angles. The Co(II) atoms reside in a pseudooctahedral environment and share the corners of a distorted cubane-like core equally with the oxygen of the phenolate group of the Schiff base ligands. The phenolate oxygen bridges three cobalt atoms, while the acetate group bridges two cobalt atoms. The distortion of the cationic unit complexes may lead to some magnetic interactions which is correlated with the small Co–O–Co angles of the top and bottom faces within the cubanes.

#### Conflict of Interests

The authors declare that there is no conflict of interests regarding the publication of this paper.

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