Synthesis, Crystal Structure of a New Co (II) Complex with 3-(4-Ethylbenzoyl) propionic Acid

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Synthesis, Crystal Structure of a New Co (II) Complex with 3-(4-Ethylbenzoyl) propionic Acid

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Abstract. A new Co(II) complex, $[CoL_2\cdot(H_2O)_4]\cdot 4H_2O$ (HL = 3-(4-ethylbenzoyl)propionic acid), has been synthesized by one-pot reaction of 3-(4-ethylbenzoyl)propionic acid, NaOH and cobalt(II) acetate tetra hydrate in CH_3CH_2OH/ H_2O (v: v = 3: 1) solution. The crystal structure of $[CoL_2\cdot(H_2O)_4]\cdot 4H_2O$ has been characterized by single crystal X-ray diffraction analysis. The result shows that the Co (II) complex belongs to monoclinic, space group $P1_{21}/n1$ with a = 9.911(2) Å, b = 9.4356(19) Å, c = 32.322(7) Å, β = 94.84(3) º, V = 3011.9(11) Å^3, Z = 4, Dc = 1.353 mg·m^{-3}, μ = 0.633 mm^{-1}, F (000) = 1300, and final R_1 = 0.0788, ωR_2 = 0.2311. The geometry around Co (II) atoms is a distorted octahedron. The Co (II) complex molecules form 2D layer structure by the intramolecular and intermolecular hydrogen bonds (O-H···O) present between the O atoms of uncoordinated and coordinated water molecules and O atoms of 3-(4-ethylbenzoyl) propionic acid.

1. Introduction

Recent years, many studies on Co (II) complexes have been reported because they show excellent properties in photoelectrocatalytic, luminescence property, magnetism, gas absorption [1-5]. Our group has been devoted to the synthesis, structure and properties of metal complexes [6-8].

In this paper, we report a new Co (II) complex from the reaction of 3-(4-ethylbenzoyl) propionic acid, NaOH and cobalt (II) acetate tetrahydrate in CH_3CH_2OH/ H_2O (v: v = 3:1) solution. The crystal structure of Co (II) complex is determined by X-ray single crystal diffraction analysis.

2. Experimental Section

2.1. Materials and Instrumentation

3-(4-Ethylbenzoyl)propionic acid (A.R.), cobalt (II) acetate tetrahydrate (A.R.), NaOH (A.R.) and solvents were commercially available reagents and used as supplied. The single crystal data of $[CoL_2\cdot(H_2O)_4]\cdot 4H_2O$ were obtained on a Bruker Smart CCD diffractometer.

2.2. Synthesis of $[CoL_2{\cdot}(H_2O)_4]{\cdot}4H_2O$

A solution of 3-(4-ethylbenzoyl)propionic acid (0.2062 g, 1.0 mmol) and NaOH (0.040 g, 1.0 mmol) in 5 mL water was added to a solution of cobalt(II) acetate tetrahydrate (0.1245 g, 0.5 mmol) in 15 mL CH_3CH_2OH with stirring. The rose Hermosa solution was stirred at 65°C for 5 hours. The suitable Co (II) complex crystals for single crystal X-ray diffraction analysis were obtained after 20 days by slow evaporation the filtrate at room temperature.

2.3. Data Collection, Structural Determination, and Refinement

A suitable crystal of Co(II) complex with dimensions of 0.19 mm × 0.18 mm × 0.16 mm was selected for data collection using Olex2 [9] on a Bruker Smart CCD diffractometer with a graphite-monochromatized MoKα (λ = 0.71073 Å) radiation. The crystal was kept at 293(2) K during data collection. The structure was solved with the ShelXT [10] structure solution program using Intrinsic Phasing and refined with the ShelXL [11] refinement package using Least Squares minimization. The molecular structure of Co (II) complex was drawn with DIAMOND [12]. Summary of crystal data for Co (II) complex are shown in Table 1.

Table 1. Summary of crystal data for Co (II) complex.

Empirical Formula	$C_{24}H_{42}CoO_{14}$
Temperature/K	293(2)
Crystal system	Monoclinic
Space group	P121/n1
a/Å	9.911(2)
b/Å	9.4356(19)
c/Å	32.322(7)
β/°	94.84(3)
Volume/Å^3	3011.9(11)
Z	4
ρ_{calc} mg/mm^3	1.353
μ/mm^{-1}	0.633
S	1.115
F(000)	1300
Index ranges	$-11 \le h \le 11$ $-11 \le k \le 11$ $-38 \le l \le 38$
Reflections collected	22999
Reflections with $I > 2\sigma(I)$	4265
Independent reflections	5297 [R(int) = 0.0424]
Data/restraints/parameters	5297/39/373
Goodness-of-fit on F^2	1.100
Final *R* indexes [$\ge 2\sigma$ (*I*)]	R_1 = 0.0788, wR_2= 0.2311
Final *R* indexes [all data]	R_1= 0.0891, wR_2= 0.2456
Largest diff. peak/hole/e Å^{-3}	1.86/−1.04

3. Results and Discussion

3.1. Structural Description of $[CoL_2{\cdot}(H_2O)_4]{\cdot}4H_2O$

Figure 1 shows the crystal structure of $[CoL_2{\cdot}(H_2O)_4]{\cdot}4H_2O$. X-ray diffraction analysis revealed that the Co (II) complex crystallizes in monoclinic system with P121/n1 space group. The Co (II) complex is composed of one Co (II) ion, two 3-(4-ethylbenzoyl) propionic acid ligands, four coordinated water

molecules and four uncoordinated water molecules. The Co (II) ion is six-coordinated with two oxygen atoms from two different 3-(4-ethylbenzoyl)propionic acid ligands and four oxygen atoms from four coordinated water molecules, respectively, giving a distorted octahedron geometry with O1, O1A, O2W and O2WA at the equatorial plane, and O1W and O1WA at the axial positions. The Co-O bond distances are 2.085(3) Å and 2.146(3) Å (Co1-O1 2.085(3) Å, Co1-O1A 2.085(3) Å, Co1-O1W 2.146(3) Å, Co1-O1WA 2.146(3) Å, Co1-O2W 2.090(3) Å, Co1-O2WA 2.090(3) Å, symmetry codes: (A) −*x*, 1−*y*, 1−*z*). The bond angles surrounding the Co (II) ion are O1-Co1-O1A = 180.0(15) °, O1-Co1-O1WA = 90.13(11)°, O1A-Co1-O1W = 90.13 (11)°, O1A-Co1-O1WA = 89.87(11)°, O1-Co1-O1W = 89.87(11)°, O1A-Co1-O2WA = 89.70(12)°, O1A-Co1-O2W = 90.30(12)°, O2WA-Co1-O2W = 180.0°, O2W-Co1-O1 = 89.70(12)°, O2WA-Co1-O1 = 90.30(12)°, O1W-Co1-O1A = 180.0°, O1W-Co1-O2WA = 90.48(12)°, O1WA-Co1-O2WA = 89.52(12)°, O1W-Co1-O2W = 89.52(12)°, O1WA-Co1-O2W = 90.48 (12)°. Figure 2 exhibits the 2D layer structure of Co(II) complex, from Figure 2, we can see that the intermolecular and intramolecular O-H···O hydrogen bonds present between the O atoms of uncoordinated and coordinated water molecules and O atoms of 3-(4-ethylbenzoyl) propionic acid play an important role in forming the 2D layer structure of Co(II) complex. The main bond lengths (Å) and angles (°) for $[CoL_2\cdot(H_2O)_4]\cdot 4H_2O$ are given in Table 2.

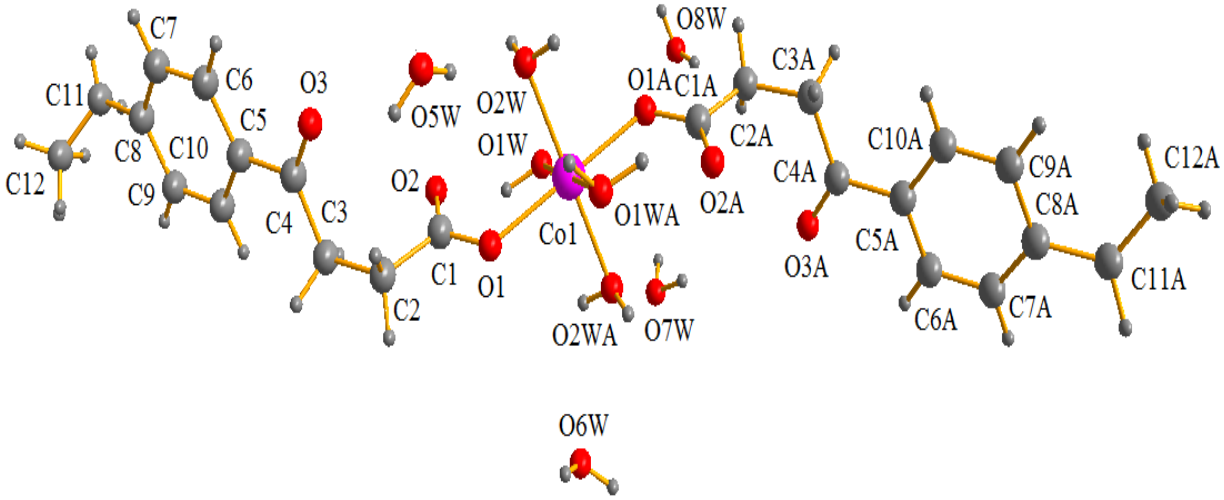

Figure 1. Asymmetric unit of the Co (II) complex with labeling scheme at 30% ellipsoidal probability. Symmetry codes: (A) −*x*, 1−*y*, 1−*z*.

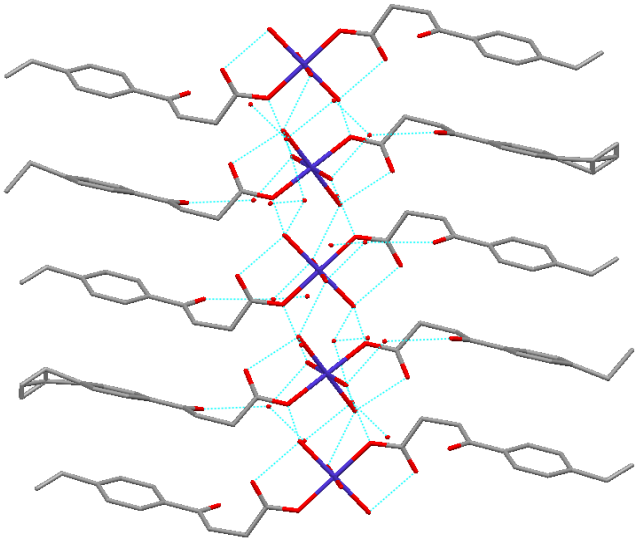

Figure 2. 2D layer structure of Co (II) complex

Table 2. Selected bond lengths (Å) and angles (°) for Co (II) complex

Bond	**Distance**	**Band**	**Distance**
Co1-O1A	2.085(3)	C3-O4	1.219(6)
Co1-O1	2.085(3)	C5-C6	1.395(6)
Co1-O1W	2.146(3)	C5-C10	1.386(6)
Co1-O1WA	2.146(3)	C6-C7	1.372(7)
Co1-O2W	2.090(3)	C8-C9	1.383(7)
Co1-O2WA	2.090(3)	C8-C11	1.581(19)
C1-O1	1.280(5)	C9-C10	1.379(6)
C1-O2	1.229(5)		
Angle	°	**Angle**	°
O1-Co1-O1A	180.0(15)	O1-Co1-O2W	89.70(12)
O1-Co1-O1WA	90.13(11)	O1-Co1-O2WA	90.30(12)
O1A-Co1-O1W	90.13(11)	O1A-Co1-O1W	180.0
O1A-Co1-O1WA	89.87(11)	O1W-Co1-O2WA	90.48(12)
O1-Co1-O1W	89.87(11)	O1WA-Co1-O2WA	89.52(12)
O1A-Co1-O2WA	89.70(12)	O1W-Co1-O2W	89.52(12)
O1A-Co1-O2W	90.30(12)	O1WA-Co1-O2W	90.48(12)
O2W-Co1-O2WA	180.0	O1-C1-C2	115.1(4)
O3-C4-C3	122.0(4)	O2-C1-O1	124.8(4)
O2-C1-C2	120.1(4)		

Symmetry codes: (A) −*x*, 1−*y*, 1−*z*.

4. Conclusion

A new Co (II) complex, $[CoL_2 \cdot (H_2O)_4] \cdot 4H_2O$, has been synthesized and structural characterized by single crystal X-ray diffraction analysis. The Co (II) complex molecules form 2D layer structure by the intramolecular and intermolecular hydrogen bonds (O-H···O) present between the O atoms of uncoordinated and coordinated water molecules and O atoms of 3-(4-ethylbenzoyl) propionic acid.

Acknowledgments

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