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Synthesis And Structure Of 5-(2-Pyridyl)Tetrazole Complex Of Co(III)

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ABSTRACT

One new Co(III) complex $\{[CoL_3](H_2O)_3\}_2$ [HL=5-(2-pyridyl)tetrazole] was prepared by hydrothermal reaction and structurally characterized. Single crystal X-ray diffraction analysis revealed that the Co(III) is six-coordinate by six nitrogen atoms of three L ligand. Crystal data: triclinic, space group P-1, α=9.493(3) Å, b=14.850(5) Å, c=15.429(5) Å, α=89.961(6)°, β=72.320(6)°, γ= 85.380(6)°, V=2064.9(11) Å^3, Z=2, D=1.754 Mgm^{-3}.

KEYWORDS

Hydrothermal synthesis;
Single crystal;
One-dimensional chain.

INTRODUCTION

In recent years, great attention has been paid to the study of metal organic frameworks not only for their fascinating structures but also for their potential applications[1]. In general, the design of appropriate organic ligands as "building blocks", together with the coordination preferences of the metal ions as "nodes", is undoubtedly the most rational synthetic strategy to produce such coordination polymers[2]. In a spontaneous self-assembly process, the structural information stored in both the organic ligand and the metal ion is read out by the coordination through their coordination geometry[3]. Therefore, it is important to choose the appropriate ligand and metal ion for constructing a specific architecture. Tetrazoles have found a wide range of applications in areas as diverse as coordination chemistry, medicinal chemistry and materials science[4-7]. 5-(2-pyridyl)tetrazole) (HL) is a multi-functional ligand having several coordination modes. Recently, the coordination chemistry of HL and its' derivatives with some metal ions, Mn(II), Zn(II), La(III), Cu(II) and even the crystal structure of HL have been studied by several groups[8-11]. Recently, we are interested in investigating the coordination chemistry of HL. However, to the best of our knowledge, the Co(III) complex with this ligand has not been reported. Herein, we report the hydrothermal synthesis and crystal structure of a new Co(III) complex.

EXPERIMENTAL

Preparation of $\{[CoL_3](H_2O)_3\}_2$

A mixture of $Co(NO_3)_2 \cdot 6H_2O$(29mg, 1mmol), HL (44mg, 3mmol) [HL=5-(2-pyridyl)tetrazole], NaOH (12mg, 3mmol) and H_2O 10mL was sealed in a 25mL stainless steel reactor with Teflon liner and directly heated to 140^{0}C for two days, then cooled to room

temperature during 12h. Block single crystals suitable for X-ray diffraction were obtained in 40% yield.

Structure determation

X-ray single-crystal diffraction data for the nickel complex was collected on a Bruker Smart 1000 CCD diffractometer at 293(2) K with Mo-Kα radiation (λ=0.71073 Å) by ω scan mode. The program SAINT[12] was used for integration of the diffraction profiles. Semiempirical absorption corrections were applied using SADABS program. All the structures were solved by direct methods using the SHELXS program of the SHELXTL package and refined by full-matrix least-squares methods with SHELXL[13]. Metal atoms in each complex were located from the e-maps and other non-hydrogen atoms were located in successive difference fourier syntheses and refined with anisotropic thermal parameters on F^2. Hydrogen atoms of carbon were included in calculated positions and refined with fixed thermal parameters riding on their parent atoms. Crystallographic data and experimental details for structural analysis is summarized in TABLE 1. Selected bond lengths and angles are listed in TABLE 2.

TABLE 1 : Crystallographic data and structure refinement summary for the complex

Complex	$\{[CoL_3](H_2O)_3\}_2$		
Chemical formula	$C_{36}H_{36}N_{30}Co_2O_6$	γ/deg	85.380(6)
Formula weight	1102.71	VÅ^3	2064.9(11)
Crystal system	Triclinic	Z	2
Space group	P-1	D/g cm^{-3}	1.754
a(Å)	9.493(3)	μ/mm^{-1}	0.894
b(Å)	14.850(5)	T / K	293(2)
c(Å)	15.429(5)	R [a] / wR [b]	0.0649/ 0.1539
α/deg	89.961(6)	Total/unique /R_{int}	10388/7192/ 0.0350
β/deg	72.320(6)		

TABLE 2 : Selected bond distances (Å) and angles (deg) for the complex

Co(1)-N(1)	1.877(5)	Co(1)-N(6)	1.879(5)
Co(1)-N(15)	1.892(5)	Co(1)-N(11)	1.956(5)
Co(1)-N(5)	1.968(5)	Co(1)-N(10)	1.977(5)
N(1)-Co(1)-N(6)	90.9(2)	N(1)-Co(1)-N(15)	92.8(2)
N(6)-Co(1)-N(15)	175.5(2)	N(1)-Co(1)-N(11)	91.8(2)

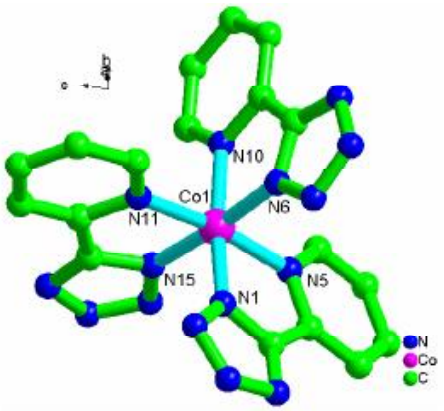

Figure 1 : View of the coordination environment of Co(III)ions in the complex

RESULTS AND DISCUSSION

The application of the hydrothermal technique to synthetic crystal growth has been used for some time[14]. The generation of coordination polymers rely on various parameters, such as the coordination mode of metal centers, different organic ligands, the ratio of metal salts and ligands, templates, solvent system, counterions, temperature, reaction time, PH values etc. The title compound was obtained as an air-stable organe solid after heated to140^{0}C for two days, and Co(II) has a process of oxidation and change to Co(III). The complex crystallizes in the triclinic symmetry with space group of P-1. X-ray single-crystal diffraction analysis reveals that it has a structure constructed from two indepentdent CoL_3 units with six water molecules. As shown in figure 1, the Co(III) locates in a distorted octahedron geometry, being coordinated with six N donors of three L ligands with the Co-N bond distances being 1.877(5) and 1.977(5)Å(TABLE 2). We have changed the M/L ratio, the reaction temperature, and added the second ligand to tune the structure of complex, only the title compound was obtained. It may attribute to the coordinate ability L ligand when the reaction is carried out in the presence of NaOH. It can coordinate to Co(III) to form the title compound and prevent other ligands to join the reaction.

REFERENCES

[1] **(a)** B.Moulton, M.J.Zaworotko; Chem.Rev., **101**, 1629 **(2001)**.
(b) M.Eddaoudi, D.B.Moler, H.Li, B.Chen, T.M. Reineke, M.O.Keeffe, O.M.Yaghi; Acc.Chem. Res., **34**, 319 **(2001)**.
[2] **(a)** W.P.Su, M.C.Hong, J.B.Weng, R.Cao, S.F.Lu; Angew.Chem.Int.Ed., **39**, 2911 **(2000)**.

[3] **(b)** W.Chen, H.M.Yuan, J.Y.Wang, Z.Y.Liu, J.J.Xu, M. Yang, J.S.Chen; J.Am.Chem.Soc., **125**, 9266 **(2003)**.
[4] J.M.Lehn; Supramolecular Chemistry-Concepts and Perspectives, VCH, Weinheim, **(1995)**.
[5] R.N.Butler; Comprehensive Heterocyclic Chemistry, ed.A.R.Katritzky, C.W.Rees, E.F.V. Scriven; Pergamon, Oxford, UK, **4**, **(1996)**.
[6] **(a)** L.Carlucci, G.Ciani, D.M.Proserpio; Angew. Chem.Int.Ed., **38**, 3488 **(1999)**.
(b) V.A.Ostrovskii, M.S.Pevzner, T.P.Kofmna, M.B.Shcherbinin, I.V.Tselinskii; Targets Heterocycl. Syst., **3**, 467 **(1999)**.
[7] S.Bhandari, M.F.Mahon, J.G.McGinley, KC.Molloy, C.E.E.Roper; J.Chem.Soc., Dalton Trans., 3425 **(1998)**.
[8] P.C.Andrews, P.C.Junk, M.Massia, M.Silbersteinb; Chem.Commun., 3317 **(2006)**.
[9] A.Facchetti, A.Abbotto, L.Beverina, S.radamante, P.Mariani, C.L.Stern, T.J.Marks, A.Vacca, G.A.Pagani; Chem.Commun., 1770 **(2004)**.
[10] H.Gallardo, E.Meyer, A.J.Bortoluzzi, F.Molin, A.S. Mangrich; Inorganica Chimica Acta, **357**, 505 **(2004)**.
[11] A.T.Rizk, C.A.Kilner, M.A.Halcrow; Cryst.Eng. Comm., **7**, 359 **(2005)**.
[12] SAINT Software Reference Manual; Bruker AXS: Madison, WI, **(1998)**.
[13] G.M.Sheldrick; SHELXTL NT, Version 5.1. Program for Solution and Refinement of Crystal Structures: University of Gottingen, Germany, **(1997)**.
[14] S.H.Feng, R.R.Xu; Acc.Chem.Res., **34**, 239 **(2004)**.