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SHORT COMMUNICATION

SYNTHESIS AND CRYSTAL STRUCTURE OF A Mg(II) COMPLEX WITH 2,6-PYRIDINEDICARBOXYLIC ACID

Xi-Shi Tai[1*], Guang-Li Wang[2], Yuan-Yuan Liu[1]

[1]Weifang University, College of Chemistry and Chemical Engineering, Weifang 261061, P. R. China
[2]Weifang Bureau of Quality and Technical Supervision, Weifang 261061, P. R. China

*Corresponding author: taixishi@lzu.edu.cn or taixs@wfu.edu.cn

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Abstract: A Mg(II) complex, $[MgL\cdot(H_2O)_3]\cdot 2H_2O$ (H_2L = 2,6-pyridinedicarboxylic acid), has been synthesized and characterized by elemental analysis, IR and X-ray single crystal diffraction analysis. The Mg(II) complex belongs to monoclinic, space group P_{21}/n with a = 8.9318(18) Å, b = 10.002(2) Å, c = 13.290(3) Å, β = 96.86(3)º, V= 1178.8(4) Å^3, Z = 4, Dc = 1.575 mg·m^{-3}, μ = 0.192 mm^{-1}, F(000) = 584, and final R_1 = 0.0349, ωR_2 = 0.1212. Structural analysis shows that the Mg(II) center is six-coordination with a NO_5 distorted octahedral coordination environment. The Mg(II) complex forms 1D chain structure by the interaction of hydrogen bonds and π-π stacking.

Keywords: *crystal structure, Mg(II) complex, 2,6-pyridinedicarboxylic acid*

INTRODUCTION

Magnesium is an indispensable element in biology because it takes part in many biochemical processes and is an essential cofactor required for the activation of a variety of enzymes [1 - 3]. Moreover, it is the metal centre of the chlorophyll which can be photosynthetic, and is related to the mechanism of some drugs [4]. So it has been regarded as the metal of life. And it is significance to study on the structure and characteristic coordination of magnesium carefully for making sure about physiological and biochemical mechanisms of all lives [5]. As part of our group to explore the synthesis and property of Mg(II) complex [6 - 9]. In this paper, a novel Mg(II) complex has been obtained and characterized by elemental analysis, IR and X-ray single crystal diffraction analysis.

EXPERIMENTAL

Materials and methods

All chemicals purchased were of reagent grade and used without further purification. Elemental analysis (C, H, N) was carried out on a Elementar Vario III EL elemental analyzer. The FT-IR spectra were obtained on a Nicolet AVATAR 360 FTIR spectrometer with KBr pellets in the range of 4000 cm^{-1}-400 cm^{-1}. The crystal data was collected on a Bruker smart CCD Area Detector.

Synthesis of the Mg(II) complex

A mixture of 2,6-pyridinedicarboxylic acid (1.0 mmol, 0.167 g), $MgCl_2 \cdot 6H_2O$ (1.0 mmol, 0.203 g) and sodium hydroxide (2.0 mmol 0.080 g) in 10 mL CH_3CH_2OH/H_2O (v:v = 1:1) was continuously stirred for 6 h at refluxing temperature. Then the mixture was cooled to room temperature, and the colourless crystals were obtained for one day. The results of the elemental analysis and IR data of Mg(II) complex are listed in Table 1.

Table 1. *Results of the elemental analysis and IR data of Mg(II) complex*

Chemical formula	Elemental analysis (%)						IR ν_{max} (cm^{-1})		
	C		H		N				
	calc.	found	calc.	found	calc.	found	ν(OH)	$\nu_{as}(COO^-)$	$\nu_s(COO^-)$
$C_7H_{13}MgNO_9$	30.05	29.78	4.65	4.52	5.01	4.89	3250	1597	1456

Crystal data and structure refinement

A colourless block single crystal with dimensions of 0.28 × 0.24 × 0.22 mm was selected for measurement. Diffraction data of the single crystal were collected by φ~ω scan mode using a graphite-monochromatic Mo $K\alpha$ radiation (λ = 0.71073 Å) at 293 (2) K on a Bruker Smart Apex CCD diffractometer. Empirical formula: $C_7H_{13}MgNO_9$; Formula weight: 279.49; Crystal system: monoclinic; Space group: P_{21}/n; a: 8.9318(18) Å; b: 10.002(2) Å; c: 13.290(3) Å; β: 96.86(3)º; V: 1178.8(4) $Å^3$; Z; 4;Dc:1.575 $mg \cdot m^{-3}$;

μ: 0.192 mm^{-1}; F(000): 584; Refl'ns collected: 7350; Independent refl'ns [R(int)]: 2817 [0.0156]; Refl'ns observed (>2δ): 2536; Refinement method: Full-matrix least-squares on F^2; Goodness-of-fit: 1.068; Final R indices [I > 2δ(I)]: 0.0349, 0.1212; R indices (all data): 0.0380, 0.1259; Limiting indices: -11 ≤ h ≤ 11,-12 ≤ k ≤ 12,-17 ≤ l ≤ 9; Min. and max. resd. dens. (e/Å^3): 0.335, 0.382; The program packages of SHELXL-97 and SHELXTL-97 were used to refine structure [10, 11].

RESULTS AND DISCUSSION

The crystal structure of the Mg(II) complex is shown in Figure 1. From Figure 1, it can be seen that the Mg(II) ion is six-coordinated by two oxygen atoms (O2, O6) and one nitrogen atom (N1) from 2,6-pyridinedicarboxylate anion, and three oxygen atoms (O3, O4, O5) from the three coordinated water molecule. The Mg(II) ion is located in a distorted octahedral coordination environment. In the coordination polyhedral structure ($MgNO_5$) of Mg(II) ion, N1, O2, O6, and O5 locate at the equatorial positions, while O3 and O4 locate at the axial positions. The complex molecule forms 1D chain structure through hydrogen bonds (Figure 2 and Table 2). The bond lengths of Mg-O and Mg-N in this work are similar to the Mg-O and Mg-N reported previously [12, 13].

Selected bonds: Mg1-O5 2.0084(10) Å; Mg1-O4 2.0321(11) Å; Mg1-O3 2.0537(10) Å; Mg1-O2 2.1565(9) Å; Mg1-O6 2.1769(9) Å; Mg1-N1 2.1051(10) Å; C1-O6 1.2635(14) Å; C2-N1 1.3266(14) Å; C6-N1 1.3329(14) Å; C7-O2 1.2624(14) Å; C1-O7 1.2447(14) Å; C7-O1 1.2459(14) Å;

Selected angles: O5-Mg1-O4 88.60(5)°; O5-Mg1-O3 87.57(5)°; O3-Mg1-O4 175.78(4)°; O5-Mg1-N1 174.97(4)°; N1-Mg1-O4 89.94(5)°; O3-Mg1-N1 94.02(4)°; O5-Mg1-O2 100.96(5)°; O2-Mg1-O4 89.96(4)°; O3-Mg1-O2 92.51(4)°; O2-Mg1-N1 74.22(4)°; O5-Mg1-O6 111.01(5)°; O6-Mg1-O4 90.88(5)°; O3-Mg1-O6 88.85(4)°; O6-Mg1-N1 73.82(4)°; O2-Mg1-O6 148.03(3)°;

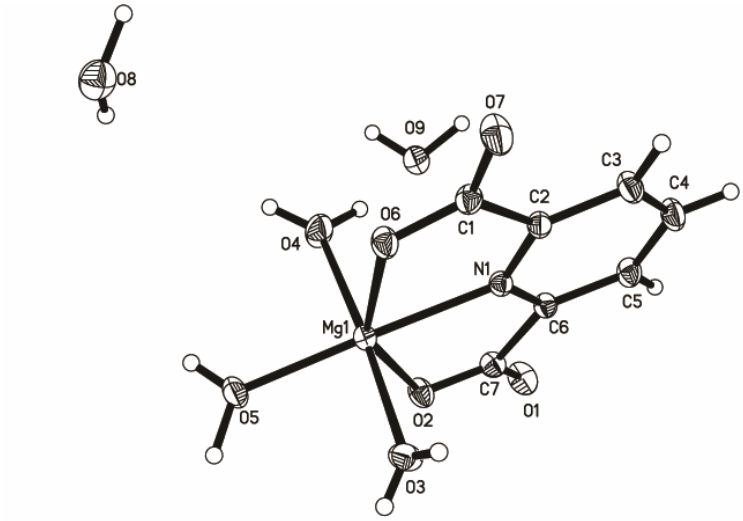

Figure 1. *Molecular structure of the Mg(II) complex, where the thermal ellipsoids were drawn at 30 % possibility*

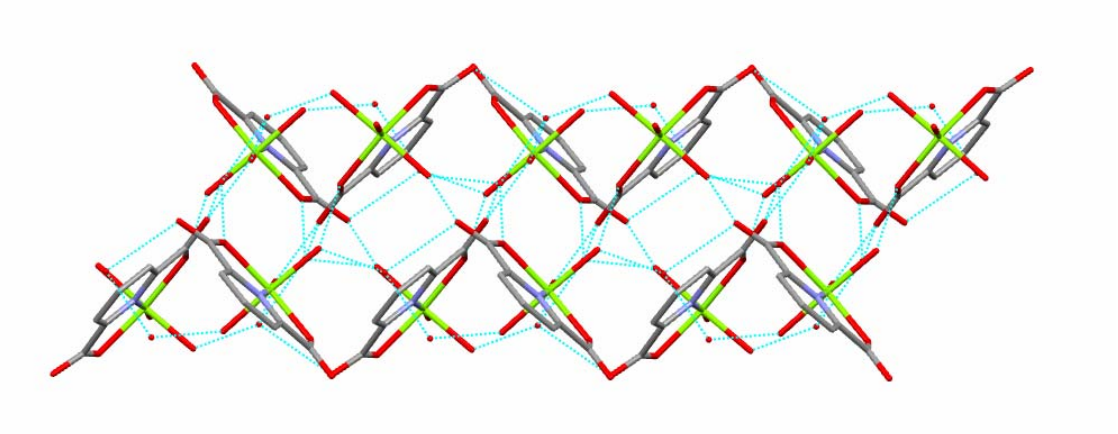

Figure 2. *One dimensional chain structure of the Mg(II) complex formed by hydrogen bonds*

Table 2. *Hydrogen bonding interactions in the Mg(II) complex*

D-H…A	*d*(D-H) [Å]	*d*(H…A) [Å]	*d*(D…A) [Å]	D-H…A [°]	Symmetry codes
O3-H3A…O8	0.8418	1.9687	2.8092	176.18	-1/2+*x*,1/2-*y*, -1/2+*z*
O3-H3C…O6	0.7235	2.0251	2.7478	176.92	1/2-*x*, 1/2+*y*, 1/2-*z*
O4-H4A…O1	0.8496	1.8226	2.6716	177.42	3/2-*x*,-1/2+*y*, 1/2-*z*
O5-H5A…O7	0.9569	1.7141	2.6689	175.38	1/2-*x*, 1/2+*y*, 1/2-*z*
O8-H8A…O1	0.8309	2.0809	2.8526	154.31	-1/2+*x*,1/2-*y*, 1/2+*z*
O9-H9A…O2	0.8462	2.0005	2.8160	161.55	3/2-*x*,-1/2+*y*, 1/2-*z*
O9-H9B…O7	0.9716	1.7648	2.7349	176.03	1-*x*,-*y*,-*z*

CONCLUSIONS

The crystal structure of $[MgL\cdot(H_2O)_3]\cdot 2H_2O$ shows that the Mg(II) center is six-coordination with a NO_5 distorted octahedral coordination environment. The Mg(II) complex forms 1D chain structure by the interaction of hydrogen bonds.

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