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SHORT COMMUNICATION

SYNTHESIS AND CRYSTAL STRUCTURE OF A NA(I) COMPLEX WITH 4,4'-BIPYRIDINE AND 2-FORMYL-BENZENESULFONATE-HYDRAZINE

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Abstract: A Na(I) complex, [Na(4,4'-bipyridine)$_2$·(H$_2$O)$_4$]·L·OH·2H$_2$O (L = 2-formyl-benzenesulfonate-hydrazine), has been synthesized. And its structure was determined by X-ray single crystal diffraction analysis. The Na(I) complex belongs to orthorhombic, space group *C*2221 with *a* = 7.9162(16) Å, *b* = 18.451(4) Å, *c* = 26.397(5) Å, *V*= 3855.7(13) Å^3, *Z* = 4, *D*c = 1.394 mg·m^{-3}, μ = 0.218 mm^{-1}, *F*(000) =1689, and final R_1 = 0.0683, ωR_2 = 0.2017. The result shows that the Na(I) center is six-coordination with a N_2O_4 distorted octahedral coordination environment. The Na(I) complex forms 1D chain structure by the π-π stacking interaction.

Keywords: *4,4'-bipyridine, 2-formyl-benzenesulfonate-hydrazine, Na(I) complex, synthesis, structural characterization*

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INTRODUCTION

The design and synthesis of schiff base ligands and their complexes are a remarkable area in coordination chemistry, because they have diverse structural flexibilities and potential applications in catalysis, electrochemistry, luminescence and medicine [1 - 5]. As a part of our group to explore the synthesis and property of schiff base ligand and their complex [6 - 9]. In this paper, we synthesized a novel Na(I) complex and determined its crystal structure by X-ray single crystal diffraction analysis.

EXPERIMENTAL

Materials and methods

2-Formyl-benzenesulfonic acid sodium-hydrazine was synthesized according to the literature [10]. 4,4'-bipyridine and other solvents 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 crystal data was collected on a Bruker smart CCD Area Detector.

Synthesis of the Na(I) complex

A mixture of 2-formyl-benzenesulfonic acid sodium-hydrazine (1.0 mmol, 0.407 g) and 4,4'-bipyridine (2.0 mmol 0.312 g) in 10 mL CH_3CH_2OH/H_2O (v : v = 2 : 1) was continuously stirred for 4 h at refluxing temperature. Then the mixture was cooled to room temperature. By evaporation the filtrate, the single crystals were obtained for 10 days. Elementary analysis: calcd for $C_{34}H_{37}N_6NaO_{12}S_2$: C, 50.44; H, 4.57; N, 10.39 %; found: C, 50.21; H, 4.20; N, 10.09 %.

Crystal data and structure refinement

A colourless block single crystal with dimensions of 0.30 × 0.28 × 0.26 mm was placed on a glass fiber and mounted on a CCD area detector. Diffraction data 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: $C_{34}H_{37}N_6NaO_{12}S_2$; Formula weight: 808.81; Crystal system: orthorhombic; Space group: *C*2221; *a*: 7.9162(16) Å; *b*: 18.451(4) Å; *c*: 26.397(5) Å; *V*: 3855.7(13) Å^3; *Z*= 4; *D*c: 1.394 mg·m^{-3}; μ: 0.218 mm^{-1}; *F*(000): 1689; Refl'ns collected: 12338; Independent refl'ns [R(int)]: 4685 [0.0258]; Refl'ns observed (>2δ): 4036; Refinement method: Full-matrix least-squares on F^2; Goodness-of-fit: 1.062; Final R indices [I > 2δ(I)]: 0.0683, 0.2017; R indices (all data): 0.0774, 0.2129; Limiting indices: $-10 \le h \le 10$,$-24 \le k \le 23$,$-35 \le l \le 26$; Min. and max. resd. dens. (e/Å^3): 0.746, -0.953; The program packages of SHELXL-97 and SHELXTL-97 were used to refine structure [11, 12].

RESULTS AND DISCUSSION

The crystal structure of the Na(I) complex is shown in Figure 1. As depicted in Figure 1, the complex molecule was made up of Na(4,4'-bipyridine)$_2$·$(H_2O)_4$, one free 2-formyl-benzenesulfonate-hydrazine ligand, one free hydroxyl and two free water molecules. The coordination environment of the Na(I) ion is six-coordination with four oxygen atoms (O5A, O6A, O6, O7A) from the coordinated water molecule and two nitrogen atoms (N2, N2A) from 4,4'-bipyridine ligand, making up a distorted forming up a distorted octahedral. In the coordination polyhedral structure (NaN_2O_4) of Na(I) ion, O5A, O6A, O6 and O7A locate at the equatorial positions, while N2 and N2A locate at the axial positions. The complex molecule forms 1D chain structure through π-π stacking interactions (Figure 2).

Selected bonds: Na1-O7A 1.934(10) Å; Na1-O6 2.049(6) Å; Na1-O6A 2.049(6) Å; Na1-O5A 2.172(9) Å; Na1-N2 2.238(3) Å; Na1-N2A 2.238(3) Å; S1-O3 1.442(2) Å; S1-O2 1.449(3) Å; S1-O1 1.452(3) Å; C17-N3 1.277(4) Å; C1-N2 1.342(6) Å; N3-N3A 1.414(5) Å;

Selected angles: O7A-Na1-O6 91.8(2)°; O6A-Na1-O6 176.4(4)°; O6A-Na1-O5A 88.22(19)°; O6-Na1-O5A 88.2(2)°; N2-Na1-O7A 91.20(12)°; O6A-Na1-N2 87.74(17)°; O6-Na1-N2 92.19(17)°; O5A-Na1-N2 88.80(12)°; O7A-Na1-N2A 91.20(12)°; O6A-Na1-N2A 92.19(17)°; N2A-Na1-O6 87.74(17)°; O5A-Na1-N2A 88.80(12)°; N2-Na1-N2A 177.6(2)°; O3-S1-O2 112.9(2)°; O3-S1-O1 114.73(19)°; O1-S1-O2 111.4(2)°; O3-S1-C14 105.72(15)°; C17-N3-N3A 111.7(3)°;

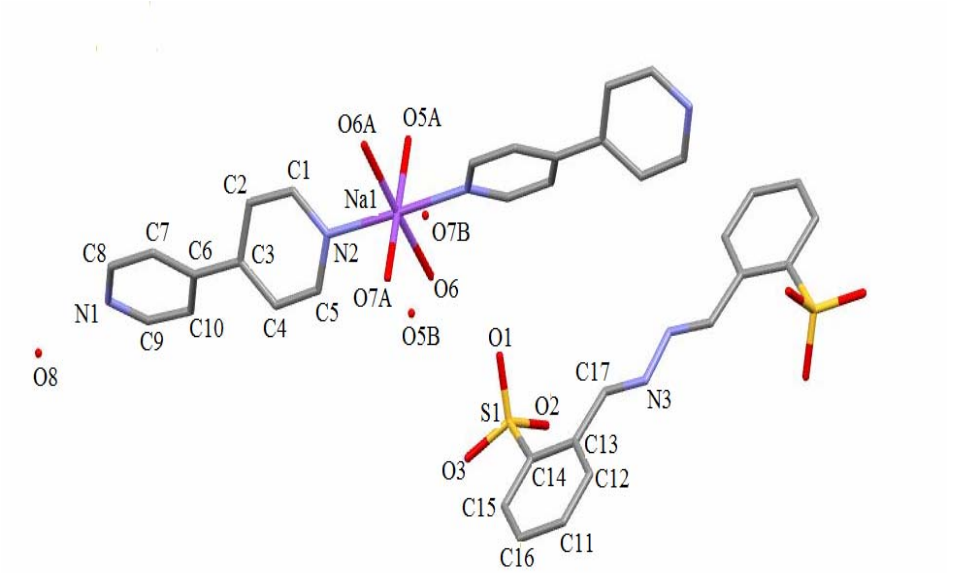

Figure 1. *Molecular structure of the Na(I) complex, where the thermal ellipsoids were drawn at 30% possibility*

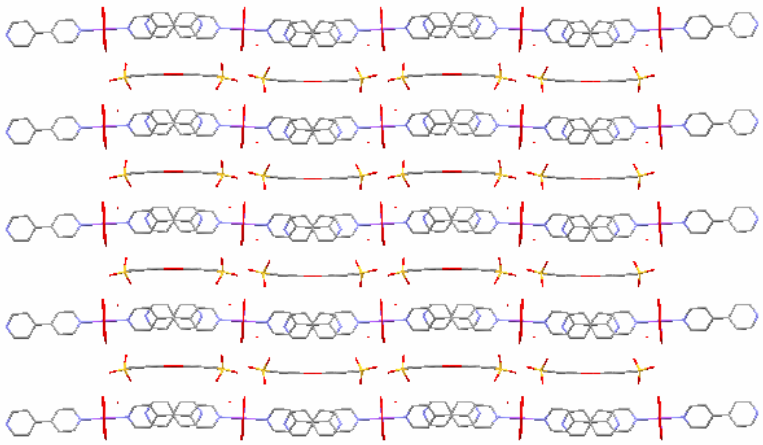

Figure 2. *One dimensional chain structure of the Na(I) complex*

CONCLUSIONS

The crystal structure of [Na(4,4'-bipyridine)$_2$·(H$_2$O)$_4$]·L·OH·2H$_2$O shows that the Na(I) center is six-coordination with a N$_2$O$_4$ distorted octahedral coordination environment. The Na(I) complex forms 1D chain structure by the π-π stacking interaction.

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